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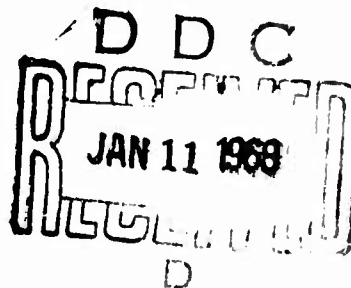| AUTHORITY |
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AFML-TR-67-330

AD824888

MAGNESIUM-LITHIUM ALLOY CASTING DEVELOPMENT

T. G. Byrer,
R. J. Jackson, and
P. D. Frost

BATTELLE MEMORIAL INSTITUTE
Columbus Laboratories

Technical Report AFML-TR-67-330
December 1967

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JAN 11 1968

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AIR FORCE MATERIALS LABORATORY
RESEARCH AND TECHNOLOGY DIVISION
AIR FORCE SYSTEMS COMMAND
WRIGHT-PATTERSON AIR FORCE BASE, OHIO

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MAGNESIUM-LITHIUM ALLOY CASTING DEVELOPMENT

T. G. Byrer, R. J. Jackson,
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FOREWORD

This Final Technical Report covers all work performed under Contract AF 33(615)-5172 from 1 June 1966 to 31 October 1967.

This contract with Battelle Memorial Institute of Columbus, Ohio, was initiated under Manufacturing Methods Project 9-164, BPSN 66-139999-2001-9164-56. It was administered under the technical direction of Mr. W. T. O'Hara of the Metallurgical Processing Branch (MATB), Manufacturing Technology Division, Air Force Materials Laboratory, Wright-Patterson Air Force Base, Ohio.

This program has been carried out under the supervision of Mr. Paul D. Frost. Mr. T. G. Byrer, Associate Chief, Metalworking Division, was the project engineer. Others participating in the program at Battelle were Mr. A. A. Popoff and Mr. R. R. Kaiser, Metalworking Division; Mr. J. Varga, Mr. C. E. Rowe, and Mr. H. E. Priest, Process Metallurgy Division; Mr. F. B. Simmons, Department of Physical and Process Metallurgy, Mr. R. J. Jackson, Department of Physical and Process Metallurgy, who prepared the state-of-the-art survey report; and Mr. F. H. Haynie and Mr. W. K. Boyd, Corrosion Division, Department of Chemistry and Chemical Engineering.

Battelle appreciates and acknowledges the cooperation of the following firms and individuals outside of Battelle who also contributed to the work on this project:

Brooks & Perkins, Incorporated
C. B. Bartholomew, A. Richards

IBM, Federal Systems Division
Dr. J. L. Solomon, R. Gridley

Dow Chemical Company
T. E. Leontis, J. N. Reding

Lithium Corporation of America
M. G. Herre, O. M. Peterson

Foote Mineral Company
L. Richardson, E. Eisworth

Lockheed Missiles & Space Company
E. Willner, Dr. J. C. McDonald

This project was conducted as part of the Air Force Manufacturing Methods Program, the primary objective of which is to develop, on a timely basis, manufacturing processes, techniques, and equipment for use in economical production of USAF materials and components. The program encompassed the following technical areas:

Metallurgy - Rolling, Forging, Extruding, Casting, Fiber, Powder
Chemical - Propellant, Coating, Ceramic, Graphite, Nonmetallics
Electronic - Solid State, Materials and Special Techniques, Thermionics
Fabrication - Forming, Materials Removal, Joining, Components.

Suggestions concerning additional Manufacturing-Methods development required on this or other subjects will be appreciated.

This technical report has been reviewed and is approved.

HENRY A. JOHNSON, Chief
Metallurgical Processing Branch
Manufacturing Technology Division

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ABSTRACT

The purpose of this program was to develop processing techniques for melting and casting Mg-Li alloys which would be adaptable to ingots of commercial sizes and applicable for converting them into plate and sheet. A state-of-the-art report on melting, casting, and fabrication procedures for Mg-Li alloys was prepared.

Melting and casting techniques were successfully developed for the production of 50- and 100-pound ingots of LA141 alloy using a vacuum-induction melting unit under an argon atmosphere of 1-inch pressure. Subsequent evaluations of this material which included various mechanical-property determinations, ultrasonic inspection, corrosion tests, and metallographic examination, indicated it to be of low impurity content and to have good mechanical properties and formability. Those studies show that high-quality Mg-Li alloy castings can be consistently produced using techniques readily applicable to existing commercial vacuum melting equipment.

Studies of fabrication procedures showed that with high quality material, processing conditions and fabrication temperatures could vary without deleterious effects on material properties or yield. However, the standard -T7 heat treatment now used as a stabilization treatment for commercial mill products can result in subsequent hardening due to room-temperature aging. Reduction of the stabilization temperature is recommended.

Studies of flux-melting techniques for the reclamation of scrap material have involved the casting of 6-, 20-, and 90-pound ingots of LA141 alloy through the use of newly developed flux compositions. Results were evaluated by visual appearance of each heat during melting and pouring and by extensive property evaluation of sheet rolled from cast ingots. The studies showed that good quality material could be obtained by flux-melting techniques and indicates considerable promise for reclaiming scrap material and ultimately reducing the cost of Mg-Li mill products.

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I

INTRODUCTION

Three problems exist today in the production of commercial magnesium-lithium alloy mill products. The major problem is one of quality. Dross, porosity, and laminations are continuing problems in commercial mill products. A second problem, which is related to the first, is that of inconsistent properties and formability characteristics plus the occurrence of microcracks in sheet material. This has led to forming and material-rejection problems in certain aerospace plants currently using these alloys. The third problem, which significantly affects the price schedule for Mg-Li alloy mill products, is the inability to recycle a majority of the melt-shop and mill scrap into the melting operation. It is estimated that unrecoverable scrap may total as much as 50 percent of the metal weight charged. This high scrap loss further increases the cost of an alloy that is already expensive by virtue of (1) the lithium cost and (2) the need for extreme care throughout the mill processing.

In view of the need for improving the producibility and availability of high-quality Mg-Li alloy mill products to meet specific aerospace requirements, the Air Force entered into a contract with Battelle to develop optimum processing techniques for melting and casting high-purity magnesium-lithium alloy ingots and for converting them into plate and sheet.

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II

SUMMARY

The objective of this program was to improve the overall quality of Mg-Li alloy mill products through the development of melting, casting, and fabrication procedures applicable to commercial-size operations. In Phase I, a state-of-the-art survey of casting and fabrication procedures for Mg-Li alloys was conducted and is included in Appendix I. The experimental program has basically consisted of (1) the casting of 50- and 100- pound ingots of LA141 alloy by vacuum-induction-melting techniques, (2) the examination of fabrication procedures for the LA141 alloy, and (3) the use of flux-melting techniques as a means of recovering metal from drossy scrap.

The melting and casting of Mg-Li alloys in a vacuum-induction unit under a reduced pressure-inert gas atmosphere was investigated in previous studies of this alloy system, and was developed further in this program as the best approach to a commercial casting technique. Melting and casting equipment consisted of a 100-pound-capacity steel crucible and tapered steel mold with heavy copper-base and copper-wall plates to promote directional solidification.

Four 50-pound heats of LA141 alloy were cast in Phase I to investigate melting and casting procedures, system pressure requirements, equipment design, and effects of charge-material quality on cast-ingot quality. The cast ingots weighed 46 to 49 pounds (92 to 98 percent yield), showed only minimal shrinkage pipe, and showed good internal soundness as measured by radiographic studies.

The results of this investigation of the vacuum-induction technique showed:

(1) The use of liquid argon at a system pressure of 1 inch of mercury minimized dross formation during meltdown and prevented fuming.

(2) The wrapping of the lithium metal in aluminum foil under a protective atmosphere prevented the formation of reaction products on the lithium and eliminated much of the dross otherwise present during melting.

(3) The use of a hinged, perforated plate provided for stirring the melt with only minimum mixing of the dross and metal during stirring.

(4) The use of a dross-retainer plate in the crucible prevented transfer of the dross layer into the mold during the pour.

Phase II studies were subsequently initiated to extend the development of melting, casting, and fabrication techniques to 100-pound heats of LA141 alloy. Two 100-pound heats of LA141 alloy were melted and cast using techniques identical to those developed for 50-pound heats. Both ingots were cast without difficulty, although operational difficulties caused the second casting to be of poorer quality than the first heat cast.

All evaluations of the first 100-pound heat including radiographs, ultrasonic inspections, microstructure analysis, mechanical-property determinations, and bend-test data indicated that this material was of quality equal to or better than material cast in Phase I. As a result of the success in defining process conditions for casting high-quality material,

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the research program was modified to require that a minimum of two (instead of six) 100-pound heats of LA141 would be cast. All studies on the LAZ933 alloy were deleted since this alloy has found little commercial use.

Included in the quality evaluations were ultrasonic material inspection of approximately 75 pounds of rolled plate by Brooks and Perkins and the IBM Federal Systems Division. These evaluations verified Battelle's conclusions that high-quality material was being produced by the vacuum-induction-melting technique. The ultrasonic inspections showed that quality of this material was as good as and generally superior to the best material commercially available today.

On the basis of these studies it is concluded that the vacuum-induction melting and casting technique will provide high quality LA141 alloy ingot using techniques directly applicable to commercial production procedures.

It was the objective of the fabrication study to determine the effects of homogenizing treatments, cross rolling, and rolling temperature on the properties of rolled sheet as measured by mechanical-property determinations, bend tests, and metallographic examination. For this work, ingot sections were forged to rolling slabs after homogenizing treatments of 8 to 48 hours at 500 F. Fabrication to 0.032-inch-thick sheet was completed at rolling temperatures of 350 or 500 F. Some material was cross rolled.

The results showed that:

(1) Cross-rolling effects could not be detected.

(2) Material rolled at 500 F had mechanical properties 15 percent above those of sheet rolled at 350 F after both were subsequently heat treated at 350 F for 6 hours.

(3) Tensile elongation was improved as homogenizing times were increased for material rolled at 350 F only.

(4) Bend-test results reflected only the temperature of rolling because unstabilized material (rolled at 500 F) fractured, while stabilized material (rolled at 350 F) withstood IT 90-degree bends without fracture.

Metallographic examinations showed typical sheet microstructures and indicated that generally good quality material was being produced.

A brief study was made of possible effects of lithium-depleted sheet surfaces (as produced by elevated-temperature processing) on properties and fabricability of Mg-Li alloys. Weight-loss evaluations on uncoated and surface-treated specimens and chemical analyses of surface and subsurface layers exposed to elevated temperatures for long time periods did not show evidence of severe lithium depletion. Mechanical-property evaluations showed some effects of long-time exposure at 500 F in terms of slight reductions in ductility of sheet specimens. However, normal processing procedures do not cause serious surface-depletion problems, and the use of protective coatings during fabrication is not required.

Studies of stabilization treatments on LA141 alloy sheet and cast slabs are described in this report. Based on hardness surveys and mechanical-property determinations, it is

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apparent that the commercial heat treatment of 350 F for 6 hours following the rolling operation is not a stabilizing treatment. Room-temperature age hardening will occur in materials after quenching from as low as 250 F. This hardening will continue for 300 to 400 hours at room temperature before any leveling off is evidenced.

However, material that is air cooled from 350 F will age to an apparently stable level after 72 hours at room temperature. Despite these increased hardnesses from aging, sheet ductility was not affected sufficiently to reduce formability to any significant degree in vacuum-induction melted material.

In view of the problems being encountered with formability of current commercial sheet products, it is recommended that the stabilization temperature be lowered from 350 F to 200 to 225 F with the time at temperature being correspondingly increased to 24 to 48 hours.

A third area of major effort was an investigation of flux-melting and screening or filtering, techniques for recovery of Mg-Li alloy scrap. A total of 19 heats of LA141 alloy scrap ranging in size from 3 to 80 pounds were cast using 11 different flux compositions as suggested by Battelle, Dow Chemical Company, and Foote Mineral Company. Results were evaluated by visual appearance of each heat during melting and pouring and by property evaluation of sheet rolled from the cast ingots. Property evaluations consisted of mechanical-property determinations, bend tests, microstructure examination, and corrosion tests, as well as Charpy impact tests on cast material.

Corrosion tests were performed to compare flux-melted scrap heats with material cast in the vacuum-induction unit. Thirty-day exposures of sheet specimens to 100 percent humidity at 95 F showed typical corrosion rates of 9 to 12 milligrams per square decimeter per day for both types of materials, which indicated that relatively good metal quality was being obtained by flux cleaning of scrap melts.

Several Dow/Battelle fluxes that were successfully developed had the following characteristics:

(1) During melting and casting, these fluxes performed as well as or better than the LiCl-LiF flux used successfully in previous programs.

(2) They provided good metal-flux and metal-dross separation with the result that good quality cost material was obtained.

(3) They were approximately 30 percent cheaper than the LiCl-LiF flux for an estimated usage cost of 25 cents per pound of metal remelted.

Other efforts during these studies included (1) the optimization of stirring and settling periods for 20-pounds heats using the Dow/Battelle flux material and (2) an extensive evaluation of screening materials, which would remove impurities from remelted scrap during pouring.

In addition to the flux studies, a variety of screening or filtering materials (glass and steel wool, metal screen, shot, glass beads, etc.) were investigated for screening dross during pouring. Molten metal was poured through the various screens into a book mold. An examination of ingots cast with these screening materials showed them to be

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of inferior quality due to gross impurity contents. The multiple-stream pouring which was obtained by screening resulted in excessive surface-film formation during pouring and subsequent entrapment of these oxide films in the ingots. It was concluded that careful melting practice and skimming during the pouring operation is more effective than straining the metal through screens. Therefore, the screening research was discontinued.

A 90-pound flux heat wa   ng the vacuum-induction melting equipment without the reduced-pressure ine   ere. Considerable flux entrapment occurred in casting this ingot due to opera   blems inherent in the equipment being used.

These studies in the scrap recovery of Mg-Li alloys show considerable promise for reclaiming scrap material and ultimately reducing the cost of Mg-Li mill products. Further efforts should be made to scale up the results of these studies to cast larger heats and continue development in optimizing flux compositions.

III

RECOMMENDATIONS FOR FUTURE WORK

The studies conducted on this program have demonstrated the applicability of the vacuum-induction melting process to the commercial production of high-quality Mg-Li alloy material. In turn, the study has also shown that, if high-quality material is cast, formability problems with the alloys can be largely eliminated.

In addition, the studies conducted to date on scrap recovery indicate that the use of fluxes can be successful in reclaiming scrap materials for subsequent remelting into commercial-grade ingot stock. The ability to reduce scrap losses, which now total 50 percent or more of the cast ingot weight, can have a significant effect on the future prices of Mg-Li mill products.

For the LA141 alloy and other Mg-Li alloys to realize their full potential as aircraft and aerospace materials, it is imperative that the results obtained in this program be implemented by scaling up the vacuum-induction melting and casting process to the production of commercial-size (500 to 1000 pounds) ingots. This could be done in existing equipment or in new equipment purchased for casting Mg-Li and other alloys that could benefit from the type of process used.

In addition, further studies in the reclamation of scrap should be made by scaling up the ingot sizes cast on this program. Additional flux compositions remain to be investigated as well as the casting of commercial-size ingots using reclaimed scrap castings as all or part of the starting-charge materials.

The inherent advantage in the use of Mg-Li alloys for military, aerospace, and commercial applications can only be realized if efforts to implement the results of this study are undertaken.

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GENERAL DESCRIPTION OF
MAGNESIUM-LITHIUM ALLOYS

The mechanical properties of magnesium-lithium alloys are similar to those of commercial magnesium-base alloys. Those containing less than 12 percent lithium have liquidus temperatures in the range of 1100 F to 1200 F and develop strengths and modulus values comparable with those of commercial magnesium alloys. Other properties, such as ductility, density, and formability, are determined to a large extent by the amount of lithium present.

The magnesium-lithium phase diagram is shown in Figure 1[1]*. Magnesium-rich alloys, that is, alloys containing up to about 16 percent lithium may be conveniently classified into three alloy types whose characteristics depend on the presence and distribution of the phases shown in the diagram:

Type I. Alloys containing up to 5.7 percent lithium solidify as an alpha terminal solid solution. The crystal structure is hexagonal close packed. The strength is less and the ductility greater than that of unalloyed magnesium.

Type II. This type contains from 5.7 to 10.3 percent lithium. These are two-phase alloys (alpha + beta) having somewhat lower strength than unalloyed magnesium but substantially better ductility and improved formability.

Type III. The third type of alloy, containing more than 10.3 percent lithium, solidifies in the body-centered cubic structure as a beta terminal solid solution. These binary alloys exhibit lower strengths than unalloyed magnesium but have tensile ductilities over 30 percent. In contrast to magnesium, these alloys can be fabricated at room temperature.

Aside from enhancing the formability of magnesium, lithium additions result in a substantial reduction in density. For example, binary alloys containing 10 and 20 percent lithium have theoretical densities of 1.42 and 1.20 g/cc, respectively, as compared with 1.75 g/cc for unalloyed magnesium. Commercial magnesium alloys have densities somewhat higher than that of magnesium.

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* References are given on page 105.

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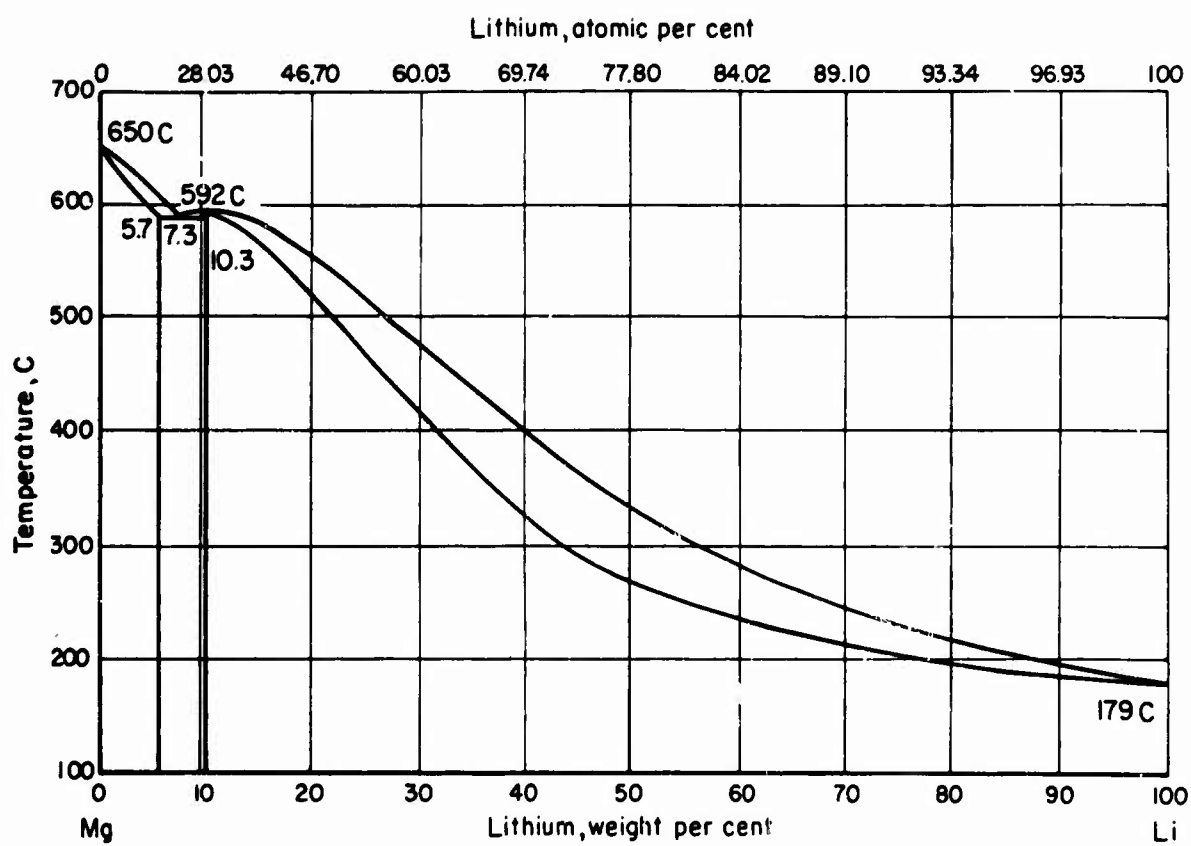
FIGURE 1.   MAGNESIUM-LITHIUM EQUILIBRIUM DIAGRAM (GRUBE)

V

BACKGROUND INFORMATION

Magnesium-lithium alloys are the lightest structural metals commercially available. The alloy LA141A (Mg-14Li-1Al) is 22 percent lighter than pure magnesium and 27 percent lighter than pure beryllium. Its density is comparable to that of most plastics but it has the stiffness (modulus of elasticity) of commercial magnesium alloys, namely 6.5×10^6 psi. This combination of low weight and stiffness has established these alloys as among the newest aerospace materials. Industry interest in the alloys for missile- and space-flight applications has come about only during the last several years, despite the fact that serious development of the alloys was started in this country as early as 1944.[2]

Like many other technical developments, the magnesium-lithium alloys were developed for a purpose that did not materialize, in this case high-strength aircraft structural applications, and they existed for a number of years before they were "discovered" by the aerospace industry.

Battelle Memorial Institute was the first organization in this country to develop these alloys and was responsible for the basic work in the development of melting techniques, forging and rolling processes, heat treatments, and actual compositions of experimental alloys, including the basic compositions of the current commercial alloys LA141, LA91, and LAZ933. This work started in 1944 and has continued, with some interruptions, to the present day under Navy and NASA sponsorship.[2,3]

The Dow Chemical Company, under Government contracts starting about 1947, continued the research and made additional significant contributions to the knowledge of the alloys. Dow, under an Army Tank Automotive Command contract, developed an armored vehicle, the M-113, having a body fabricated entirely from a magnesium-lithium alloy.[4] Dow also developed and patented a process for producing pellets or grains of the alloys and consolidating them by hot extrusion into mill shapes having superior corrosion-resistance properties.

Brooks and Perkins, now the only commercial producer of magnesium-lithium structural mill products, first made the alloys, under Battelle supervision, in about 1952. A number of heats were made and rolled for Navy armor programs. This work terminated in about 1954, and Brooks and Perkins did not reactivate it until the demand for the alloys in aerospace applications started around 1960.

In 1957 Battelle undertook for the Army Ballistic Missile Agency (ABMA), now the George C. Marshall Space Flight Center — NASA, a development contract to evaluate magnesium-lithium alloys for possible missile and space applications. The report from that work was issued in 1960.[5] It selected two alloys as having the most desirable combination of properties for aerospace uses. These were LA141 and LA91. The former contains 14 percent lithium and 1 percent aluminum; the latter contains 9 percent lithium and 1 percent aluminum. These were selected after screening a very large number of alloys that had been melted and evaluated in the early Battelle work. In the ABMA study, many alloys were rejected because, while having higher strength, they were not stable at these high strength levels. Other alloys were rejected because they had higher densities than these two. The two alloys selected are not high-strength materials, but they have a useful combination of low density, good modulus of elasticity, moderate strength, and excellent formability.

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The LAZ933 alloy (Mg-9Li-3Al-2Zn) was developed on a later program for NASA.[6] This alloy has a higher strength and strength-to-weight ratio than either the LA141 or LA91 alloy while retaining excellent formability and fabricability characteristics.

These developments in lightweight alloys attracted the attention of manufacturers of aerospace hardware, notably IBM's Federal Systems Division at Owego, New York, and the Lockheed Missiles and Space Company at Sunnyvale, California. These two organizations ordered quantities of the LA141 alloy from Brooks and Perkins and have conducted detailed, extensive programs of evaluating the alloy in sheet form for a number of space-hardware components. Lockheed has used this material in a number of electronic housing units, brackets, cable-attachment holders, and other parts for the AGENA booster and for satellite systems. Thus, components made from Mg-Li alloys are now orbiting in space inside these vehicles.

IBM has made gyro-control parts for the Gemini from the alloys, and now has a major contract from NASA to develop a computer and data analyzer — the frames for which are being made almost entirely from LA141 alloy. This system will be used in the Saturn V. Other companies have less ambitious programs with the alloys but are learning how to use them in space hardware.[3]

VI

EXPERIMENTAL RESEARCH PROGRAM

The purpose of this program was to develop processing techniques for melting and casting Mg-Li alloy ingots of commercial sizes and for converting them to plate and sheet. The overall objectives were to

(1) Improve overall quality of Mg-Li mill products

(2) Develop melting and casting procedures applicable to commercial-size heats (1000 pounds or more)

(3) Establish rolling schedules for processing commercial-size ingots (up to 500 pounds).

The experimental work program was divided into two phases. The basic objective in Phase I was to develop melting, casting, and fabrication procedures for producing high-quality Mg-Li alloy plate and sheets. Heats weighing up to 50 pounds were to be cast by the vacuum-induction-melting process, with flux-melting techniques being investigated as a means of recovering metal from drossy scrap. Fabrication procedures were to be investigated to optimize processing techniques for sheet and plate products.

Included in Phase I was a state-of-the-art survey of casting and fabrication of Mg-Li alloys. The findings of this survey are reported in the Appendix I.

Phase II represents a scale-up of the results of Phase I in the production and fabrication of 100-pound heats by the vacuum-induction-melting process. Scrap recovery studies were to be continued based on the results of the initial studies in Phase I.

VII

VACUUM-INDUCTION MELTING STUDIES

The melting and casting of Mg-Li alloys in a reduced pressure-inert atmosphere system was first explored in previous studies at Battelle,[5] and showed considerable promise. In the present study, this system was selected as the best approach to a commercial casting technique for these alloys. The basic melting and casting cycle employed previously in this technique is as follows:

(1) Charge clean, dry metal to the steel crucible mounted in the induction furnace.

(2) Evacuate chamber to below 10 microns of mercury pressure and continue pumpdown until the leak rate is maintained below 10 microns per minute.

(3) Backfill with argon to a total pressure of 50 to 100 millimeters of mercury.

(4) Induction heat the steel crucible to melt the charged material.

(5) Stir melt vigorously, allow 5 minutes for settling, reheat to temperature (1300 to 1400 F), and pour into a steel mold.

This technique is discussed in more detail in the Appendix I.

Figure 2 shows the vacuum-induction unit used in this program. This top-pouring unit, built by theConsolidated Vacuum Corporation, operates at a frequency of 1920 cps at a power input of 100 kilowatts. Up to 300 pounds of steel or about 150 pounds of magnesium can be melted in this unit.

Raw Materials

Commercial-grade materials were used in melting of Mg-Li alloys on this program. Listed below are the material forms and purity levels of each alloying element:
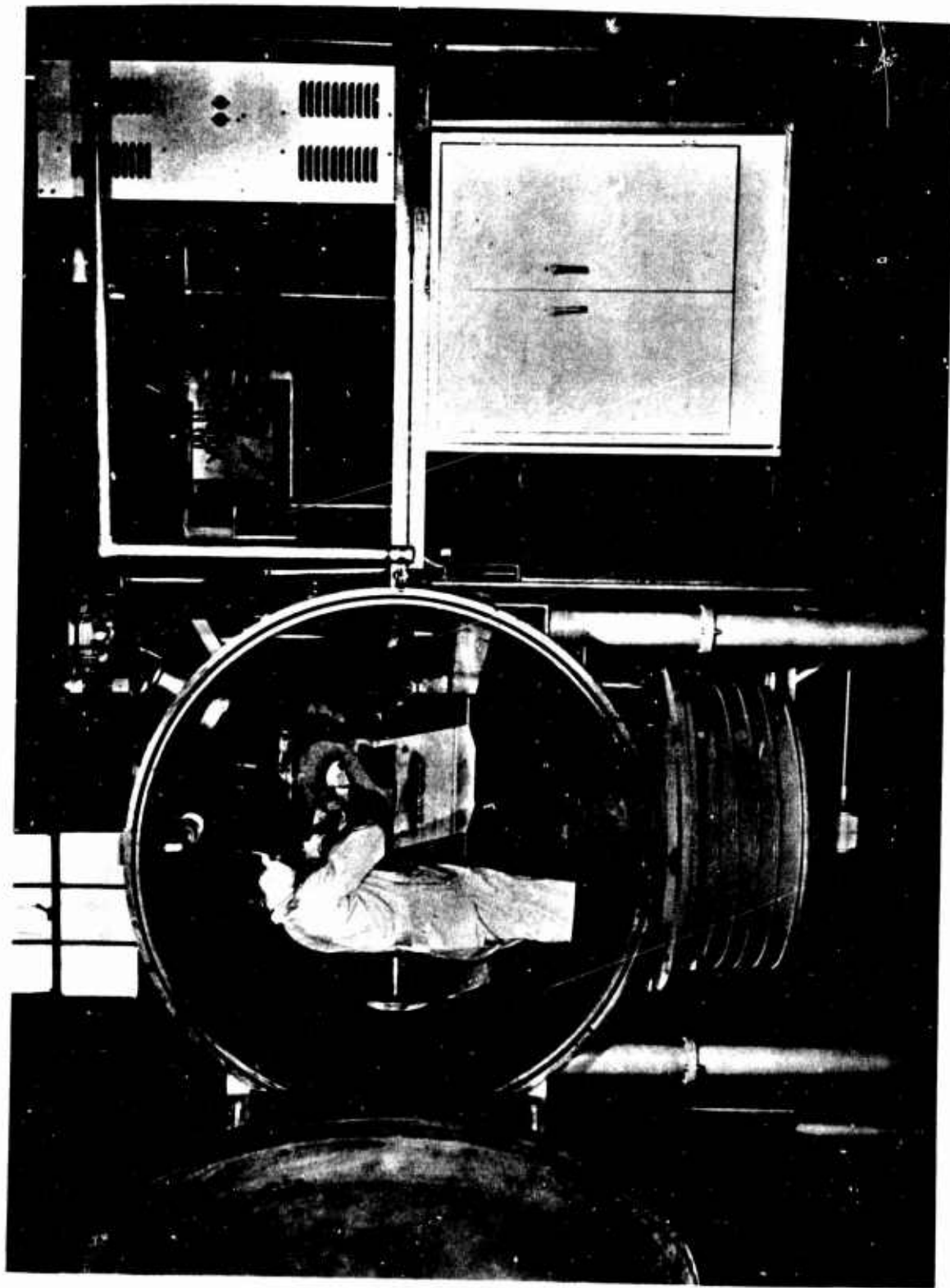
FIGURE 2.   VACUUM-INDUCTION FURNACE USED FOR MELTING AND CASTING
MAGNESIUM-LITHIUM ALLOYS

N46631

Material	Form	Source	Purity Level or Composition
Magnesium	Pig	Dow	99.98 percent Mg min
Lithium	1-pound ingots[a]	Foote	99.96 percent Li + 0.0062 percent Na + 0.0028 percent K
	1-pound ingots	Lithium Corporation of America	Not available
Aluminum	Pig	Alcoa	99.91 percent Al min + 0.032 percent Fe + 0.046 percent Si
	Foil	Reynolds	1100 Aluminum alloy contains: 0.2 percent Cu 0.05 percent Mn 0.10 percent Zn 1 percent Fe+Si
	Wire	--	99 percent Al min

(a) Experimental quantities also supplied in thin-walled aluminum cans.

Lithium Encapsulation

The previous studies involving the casting of Mg-Li alloys in a vacuum-induction system showed that dross formation was in evidence on the top of the melt even though the system was evacuated and backfilled with a partial pressure of argon. This dross apparently came largely from the exposure of lithium metal to the air during loading of the crucible. For the large heats to be made in this study, attempts were made to obtain lithium that was cast in an aluminum or magnesium can or otherwise protected from the air. This would completely eliminate the exposure of lithium to the air, reduce dross formation during melting, and subsequently reduce chances for dross entrapment during pouring.

Contacts were made with Foote Mineral Company and Lithium Corporation of America regarding the availability of encapsulated lithium. Both companies conducted experiments with magnesium as a canning material rather than aluminum. The Li:Al ratio is 14:1.5 in the LA141 alloy or (9.3 to 1), and a 1-pound ingot of lithium would require a maximum can weight of less than 0.11 pound if made of aluminum. The thin can wall required to meet this weight ratio caused difficulties because lithium alloys with the aluminum, causing melting of the can.

The magnesium can appeared more favorable since the Li:Mg ratio is reversed, 1:6, and a heavy-walled magnesium can could be used. However, pure magnesium tubing is not commercially available and the costs involved in obtaining extruded magnesium tubing and producing a sealed can would triple the lithium metal cost per pound, according to one supplier.

While these initial investigations were in progress, it was decided at Battelle to consider another procedure for protecting the lithium metal. The as-purchased cans of lithium metal were placed in a dry-box under an argon atmosphere. The cans were opened in this dry box and the 1-pound lithium ingots tightly wrapped in precut sections of commercial-grade aluminum foil. Wrapped ingots were kept in the dry box until ready to charge into the induction furnace. In this manner, the contamination of lithium was essentially eliminated during these studies.

Following this same approach, the lithium suppliers indicated that lithium ingots could be supplied in an aluminum bag which is in turn protected by a paper-plastic-aluminum laminate bag to provide long-time protection from air and moisture. None of these bags was supplied, however. Lithium Corporation did supply a few aluminum-bagged* ingots but these were packaged in air and no attempt was made to further protect the lithium in the bag. Examination of this material prior to use indicated that some localized oxidation was occurring due to pinhole porosity in the aluminum foil. Thus, some additional protection such as a laminate bag would be necessary if this technique were to be used commercially.

Unless some new developments occur in packaging techniques, the procedure described above for foil wrapping of the lithium in a drybox will be used during the remainder of the program.

Preliminary Melting Studies

Two 6-pound melts were made in the vacuum-induction unit to establish heating-cycle requirements, familiarize operating personnel with induction-melting procedures for Mg-Li alloys, and attempt to evaluate the effect of canned versus uncanned lithium in reducing dross formation during melting. The aluminum-canned material for this work was available from studies conducted in the past. In these heavy-walled cans, the Li:Al ratio is quite low (2:1), so that the actual melt composition of the first heat using two encapsulated cans was 71Mg-17.3Li-11.7Al. The second melt using uncanned lithium metal was the LA141 alloy composition.

Neither heat was poured. The system was pumped down to 10 microns pressure and backfilled with argon to a pressure of 50 millimeters before melting. After each charge was melted, the bath was stirred, heated to 1300 F, and allowed to solidify in the crucible.

Both ingots showed about equivalent amounts of dross formation. Comparisons may not be valid, however, since different lithium contents were used. In both cases, dross formation was not excessive.

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* Lithium placed between 2 sheets of aluminum foil which were then heat sealed together to form a sealed bag.

Early in the Phase II studies, Foote Mineral Company supplied several 1-pound ingots of lithium which were poured directly into thin (0.006-inch wall) aluminum alloy (3003), cans. The cans were subsequently sealed by epoxy cementing a thin foil disk on the open end of the can as shown in Figure 3.

A 15-pound heat using these canned ingots was subsequently cast to evaluate handling characteristics, melting procedures, and ultimate quality of the cast ingot.

The canned lithium ingots, along with sufficient aluminum and magnesium to provide the proper alloy composition, were melted under argon and cast in a steel mold using procedures developed in this study. The virgin magnesium and aluminum were melted initially and the aluminum-canned lithium ingots were added to the molten bath. The lithium was slowly lowered into the melt and held under the melt surface until solution occurred. The epoxy used to seal the can lid produced a dense smoke upon ignition but caused no further problems during the melting and casting cycle.

A 13.5 pound ingot was subsequently cast without difficulty. Impurity analysis of the ingot shows the following:

	Weight Percent
Mn	0.001
Fe	0.01
Co	<0.001
Ni	<0.001
Si	0.003
Cu	0.001
Ca	<0.001

The iron content is above the allowable specification limit of 0.005 percent maximum. This is prob_bly due to the high iron content (0.7 percent) present in the 3003 alloy can material used in this heat. Changes in the selection of canning material would alleviate this problem.

Based on the results of these various approaches to the problem of lithium encapsulation, it appears now that the use of an aluminum can is feasible and practical. A sealing technique for the can lid should represent no particular problem since standard techniques now used in the production of various commercially canned products could be applicable here.

No estimates are available on the ultimate cost of aluminum-canned lithium ingots. Foote Mineral Company suggests that only a very nominal cost would be incurred in canning in relation to the cost of the lithium which is quite high ($7 to 10 per pound). However, these costs should be more than offset by the increase in material yield that can be realized by improvement of cast-ingot quality.
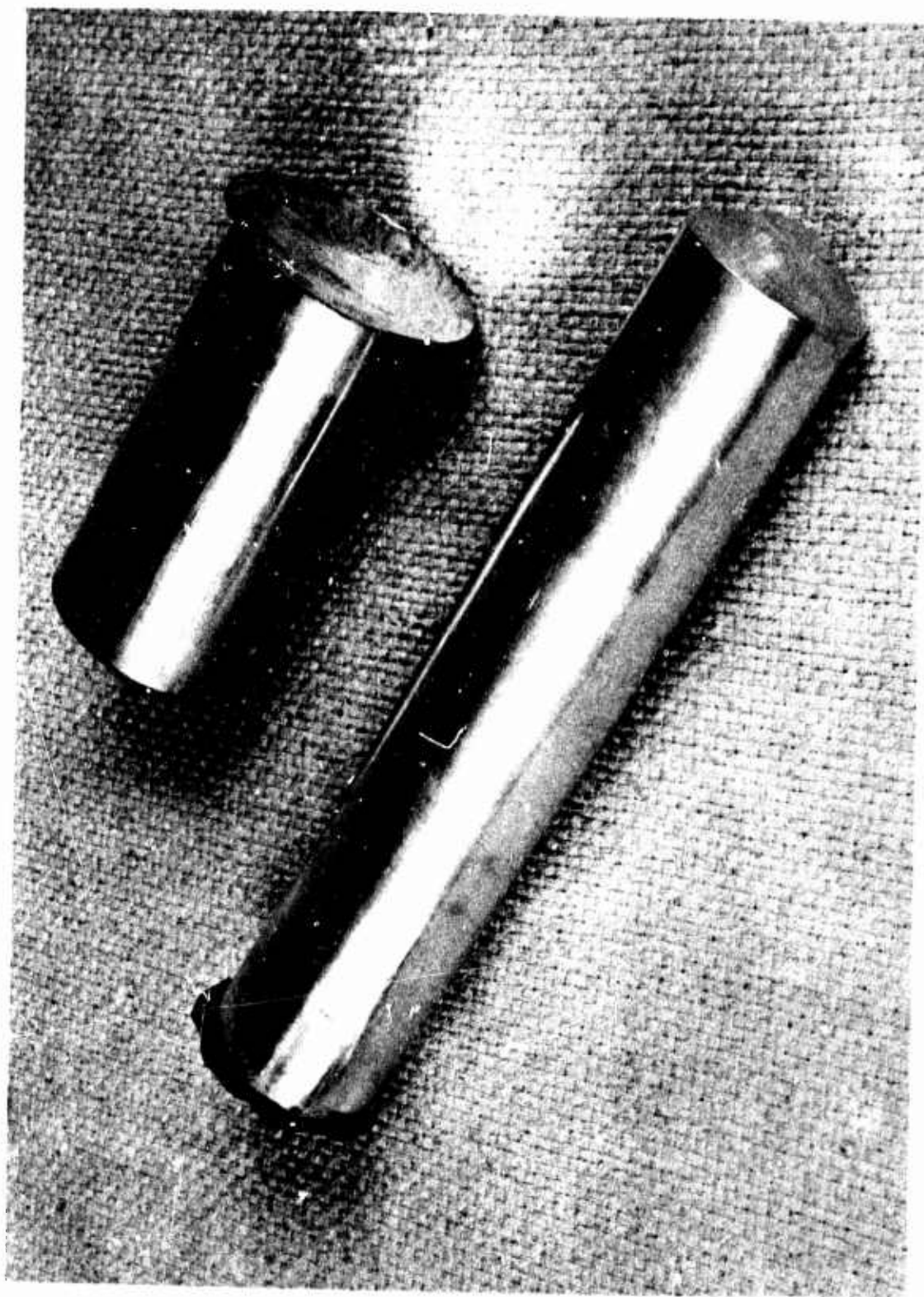
30631

FIGURE 3. ALUMINUM-CANNED LITHIUM INGOTS SUPPLIED BY FOOTE MINERAL COMPANY

Casting of 50-Pound Heats - Phase I

The basic objective in this program was to devise the optimum melting and casting technique for Mg-Li alloys that would consistently provide high-quality material for the fabrication of wrought products. The vacuum-induction melting technique described previously was to be used throughout this program as it represents the best known approach to the ultimate commercial production of Mg-Li alloys.

The work conducted previously for ABMA (now NASA) provided much of the basic development of the vacuum-induction technique. However, it was anticipated that refinements in equipment design and operating procedures would be required to optimize conditions for the melting and casting of 50- and 100-pound heats of Mg-Li alloys.

It was Battelle's opinion at the outset of these studies that two main factors would affect the ultimate quality of the cast ingots. The first concerned the material quality of the starting charge. Contaminants can come from two sources, internal or external. The control of contaminants within the charging material is assumed by using high-quality starting stock. The elimination of surface contaminants, in the case of magnesium, is obtained by wire brushing the surfaces to remove dirt and loose oxides. The use of foil-wrapped lithium as described above represented the best approach on this program for protection of lithium metal surfaces. Thus, it appeared that the first factor could be closely controlled from the outset by using high-quality materials coupled with proper metal-surface preparation and/or protection.

The second factor contributing to ingot quality is the atmosphere in the furnace during melting and casting. It is known from the previous research (and indicated in the preliminary studies reported above) that moisture present in the inert gas does contributes to dross formation on the top of the melt as well as during the pour. Thus, it was the intent of this study to use as low a system pressure as possible during the melting and casting cycle and to use as pure an inert gas as could be obtained either commercially or by special treatment of commercial gases.

Under these conditions then, the melting and casting studies in Phase I were initiated. Four 50-pound heats of LA141 alloy were cast in Phase I. The details concerning these studies are presented here.

Equipment Design

All tooling was designed to facilitate casting of 100-pound heats although only 50-pound heats were cast in Phase I. Figure 4 shows the crucible design and the initial mold design. This mold has a copper bottom and steeply tapered sides to promote directional solidification. It was hoped that this technique would prevent severe piping during solidification and preclude the need for a water-cooled base.

A second mold design as shown in Figure 5, was also used. This mold had a more conventional taper and heavy copper plates on the sides of the mold as well as on the base to promote directional solidification.

Necessary to drain crucible when mold is poured

100-lb level

50-lb level

Copper base

4°

Reversed, pyramidal mold (steel) –
Top = 18 3/8" x 18 1/8", Bottom = 7"x7",
Height = 20"

Cylindrical crucible (steel) –
12 1/4" I D x 13" O D x 28" deep.
2" x 6" cutout for pouring lip
allows 22" depth for bath

16°

20°

Angle of inclination of
mold wall at start of pouring
100-lb heat = 0°

100-lb level

50-lb level

Pivot axis
for unit

Induction melting
furnace (3000 c/sec)

① Filter basket, 100-lb heat
② Filter basket, 50-lb heat
③ P.B. sillimanite
④ Coil coating, refr. slurry
⑤ Glass cloth retainer
⑥ Ampco-Mag (or Al) backing
⑦ Asbestos board, 1/8"

Cables (2) to winch drum

1/16" = 1"

C-54303

FIGURE 4. SECTIONED VIEW OF VACUUM-INDUCTION MELTING UNIT SHOWING
MOLD AND CRUCIBLE ARRANGEMENT FOR CASTING 50- to 100-POUND
HEATS OF MAGNESIUM-LITHIUM ALLOYS

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Steel
mold

5° taper

1-inch thick
copper wall plates

2-inch thick
copper base

A 55324
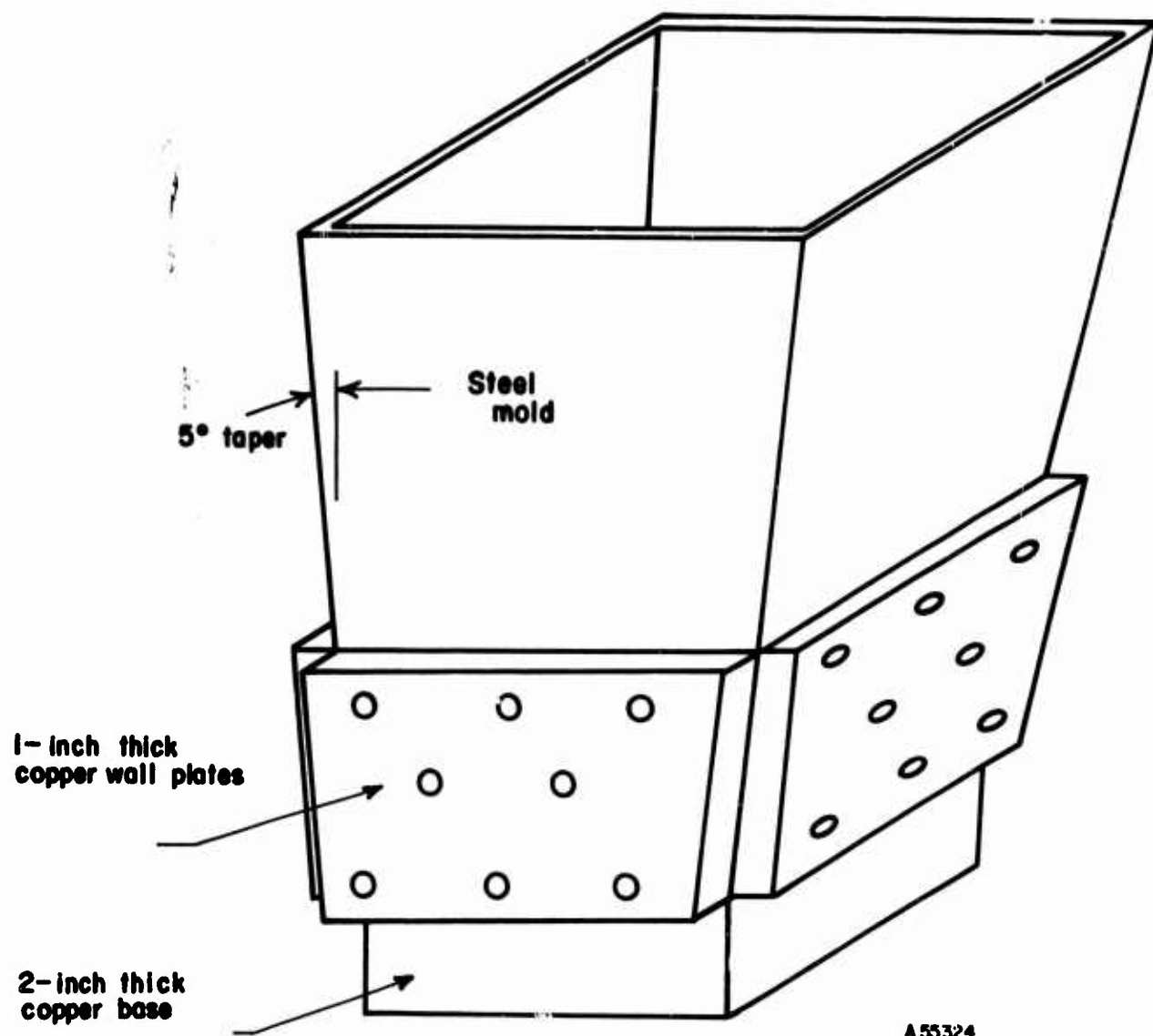
FIGURE 5. SECOND MOLD DESIGN USED FOR CASTING
50-POUND LA141 ALLOY INGOTS

Melting and Casting

Table I identifies the processing conditions for melting and casting four 50-pound heats of LA141 alloy. All lithium was foil-wrapped as described previously. Magnesium pig was sectioned and exterior surfaces of all pieces were polished with a powered wire brush. The melting and casting cycle was outlined earlier in this section of the report.

TABLE I. PROCESS CONDITIONS FOR MELTING AND CASTING
50-POUND HEATS OF LA141 ALLOY

Heat	Melt Size, lb	Aluminum Foil-Wrapped Lithium	Inert Atmosphere	System Pressure, in	Pouring Temperature, F	Cast Ingot Weight, lb
C	50	Yes[a]	Tank argon[c]	0.5	~1400[e]	46
D	50	Yes[a]	Tank argon	2.0	1330	46
E	50	Yes[b]	Liquid argon[d]	1.0	1380	47
F	50	Yes[a]	Liquid argon	1.0	1350	49

(a) Foil wrapped under argon atmosphere.
(b) Foil wrapped in air.
(c) Moisture content = 5 grains H_2O/1000 cu ft of gas.
(d) Moisture content = 2 grains H_2O/1000 cu ft of gas.
(e) Thermocouple failed; melt temperature estimated.

Heats C and D. The first two heats were cast in the mold design shown in Figure 4. The principal objectives in these initial studies were to evaluate (1) the effect of system pressure variations (0.5 inch and 2 inches mercury) on dross formation and fuming during melting and pouring and (2) the effect of the mold design on providing directional solidification. Welding-grade argon was used in both cases.

Considerable fuming occurred under 0.5 inch of mercury pressure but dross formation on the melt surface was very light. In fact, this dross layer was too thin to be continuous and the bright metal surface was visible underneath the dross. The heavy fuming also caused some difficulty in observing pouring of the ingot. However, a good cast ingot was obtained as seen in Figure 6. The radiograph shown in Figure 7 shows that shrinkage porosity was contained in the top 3 inches of the cast ingot.

The second heat was melted and cast under a system pressure of 2 inches of mercury with a noticeable decrease in fuming and a pronounced increase in dross formation. This ingot had shrinkage porosity confined to the top 2-1/2 inches of the ingot.

Chemical analysis of these two heats is listed in Table II along with the standard material specification for LA141 alloy. Analytical techniques were supplied by Brooks and Perkins. The data shows that all elements present are within specification.
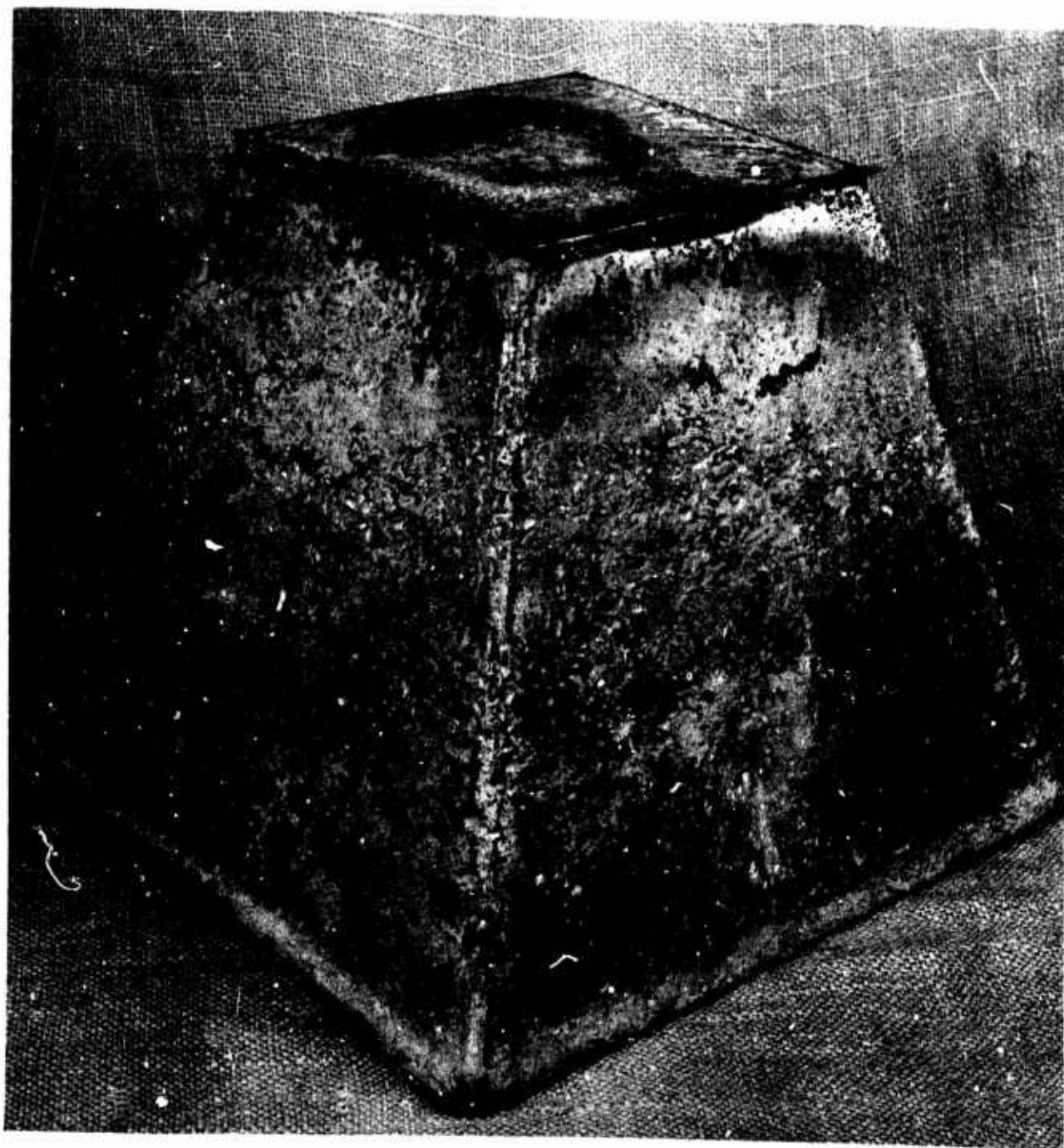
FIGURE 6. 50-POUND HEAT OF LA141 ALLOY CAST AT BATTELLE
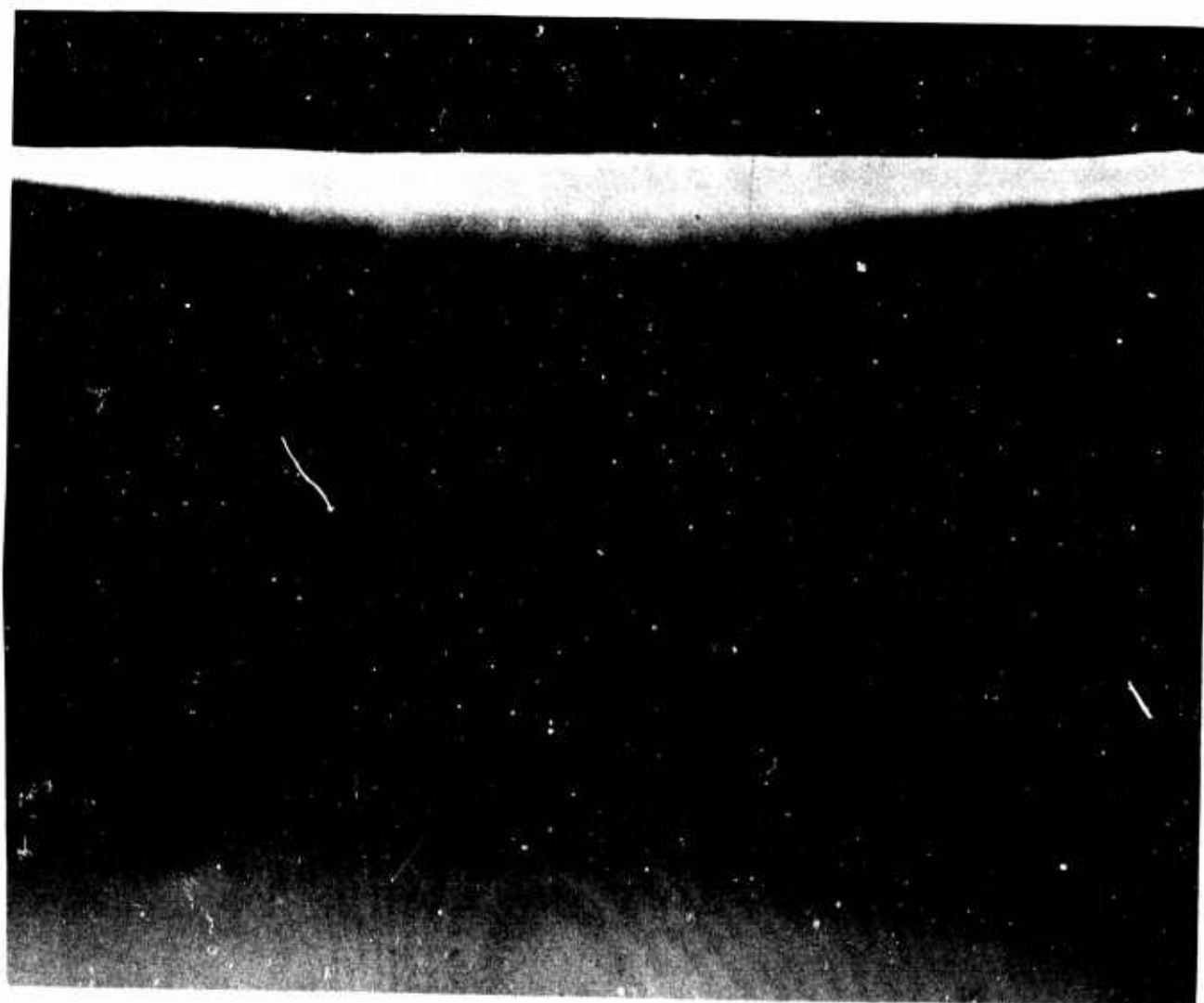
FIGURE 7. RADIOGRAPH OF THE TOP PORTION OF THE FIRST 50-POUND
CAST INGOT OF LA141 ALLOY

Shrinkage voids confined to the top 3 inches of ingot.

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TABLE II. CHEMICAL ANALYSIS OF TWO 50-POUND HEATS OF LA141 ALLOY

Heat	Chemical Composition, percent							
	Li	Al	Mn	Fe	Cu	Ni	Si	Mg
C	14.0	1.46	<0.05	0.003	0.006	<0.005	<0.01	rem.
D	14.0	1.40	<0.05	0.003	0.04	<0.005	<0.01	rem.
Commercial Specifications for LA141	13-15	1-1.5	0.15 max	0.005 max	0.04 max	0.005 max	0.10 max	rem.

The results obtained with these two heats indicated that variations in the system pressure were influencing the amount of dross formation that occurred. In addition, the initial mold design with a heavy copper bottom worked satisfactorily in promoting directional solidification, although it would be desirable to further reduce the amount of shrinkage porosity that was obtained.

Based on these results, a second series of two heats were cast.

Heats E and F. For these heats, the modified mold design shown previously in Figure 5 was used. The wall taper was reduced drastically compared to that of the first design, and heavy copper "heat sinks" were fastened to the lower portions of the mold walls to promote directional solidification.

With these two heats, the following factors were to be investigated:

(1) Effect of low-impurity liquid argon on reduction of dross forma-
tion during melting

(2) Effect of modified stirring rod and skimming plate in preventing
dross transfer from the crucible to the mold during the pour.

For both heats, system pressures of 1 inch of mercury were used. For the initial heat (Heat E), aluminum-bagged lithium supplied by the Lithium Corporation of America was used. Standard aluminum-wrapped lithium ingots were used for the second heat (Heat F).

Both heats were cast without difficulty. The system pressure of 1 inch of mercury resulted in some fuming during melting and pouring but did not hamper visibility in the chamber during the cycle. Considerable dross was formed in Heat E using the bagged lithium ingots. This was not unexpected since some air was present in the bag and some contamination would be caused by air leakage through pinholes in these foil bags. A modified stirring rod and retainer plate (described below) served to retain this dross in the crucible and prevent its transfer into the mold.

With Heat F in which liquid argon and the normal aluminum-wrapped lithium were used, dross formation was reduced to its lowest level, which indicated that the control of contamination from the lithium and the system atmosphere will minimize the amount of dross formed.

Figure 8 shows Heats E and F and the mold used for casting these ingots. The minimal shrinkage porosity in the ingot top is evident in Figure 8 and in the radiograph of Heat F in Figure 9.

These ingots were sectioned and fabricated to plate for ultrasonic testing by Brooks and Perkins and by IBM's Federal Systems Division. The results of the ultrasonic inspection, detailed in a subsequent section of the report, indicated that the material supplied for evaluation was of better quality than that required in the current Class C inspection specification setup for Mg-Li alloy mill products.

The combination of proper system atmosphere, the protection of lithium prior to melt down, and the design of stirring and skimming equipment produced the highest quality material cast to date and indicated that plans could proceed immediately for the casting of 100-pound heats.

Stirrer and Retainer-Plate Design

The successful separation of dross and metal in Heats E and F can be credited to the stirring and skimming techniques mentioned above. Figure 10 shows the stirring disk used in Heats C and D and the redesigned stirrer and dross-retaining plate used in Heats E and F.

With the hinged stirring disk, the disk can be inserted into the melt at 90 degrees to the melt surface to prevent forcing dross down into the melt and then opened to provide stirring. The extraction of this disk subsequently serves to lift some dross off the melt surface and trap it on the disk. The dross-retaining plate was used in previous studies and provides for a "bottom-type pour" to keep dross from entering the mold.

These techniques, resulting in the casting of high-quality ingots, were used again for 100-pound heats.

Casting of 100-Pound Heats - Phase II

The process conditions developed in Phase I were used in the casting of 100-pound heats in Phase II. All lithium was wrapped in aluminum foil while under an argon atmosphere. This technique prevents the reaction that occurs when lithium is exposed to air and the oxide and/or nitride that will rapidly form as a result of this reaction.

In Phase I studies, the magnesium pig used for melting was sectioned and all exterior surfaces polished with a powered wire brush to remove all loose surface oxides that might be present. To expedite this operation during the present studies, the magnesium sections were given a light chemical pickle in a 2 percent solution of sulfuric acid. This "bright dip" operation quickly removed most surface oxides.

Melting and Casting

Table III describes the processing conditions for melting and casting Heats G and H. The melting and casting cycle used is outlined below:
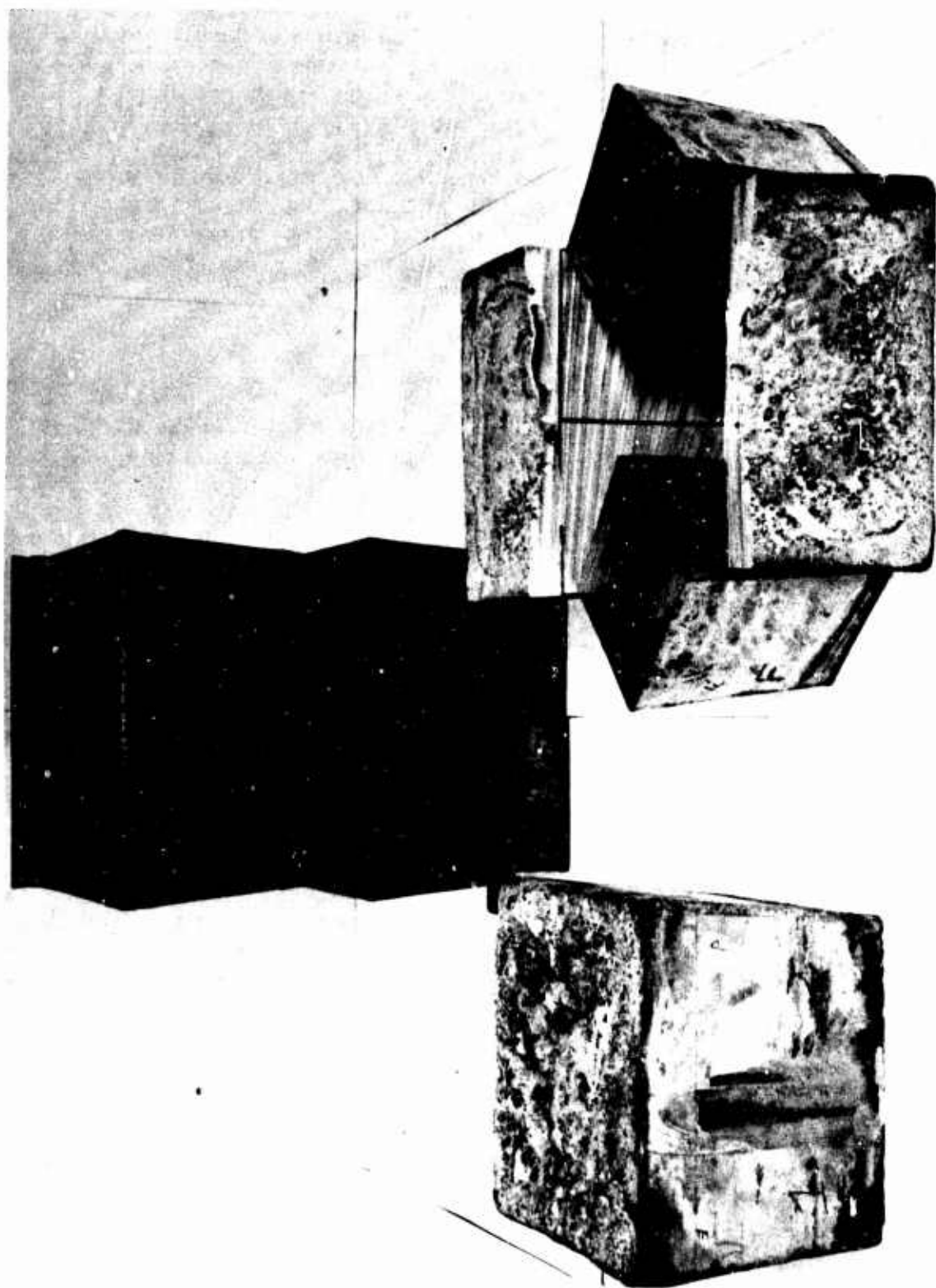
FIGURE 8. TWO 50-POUND INGOTS OF LA141 ALLOY AND INGOT MOLD USED IN CASTING THESE INGOTS
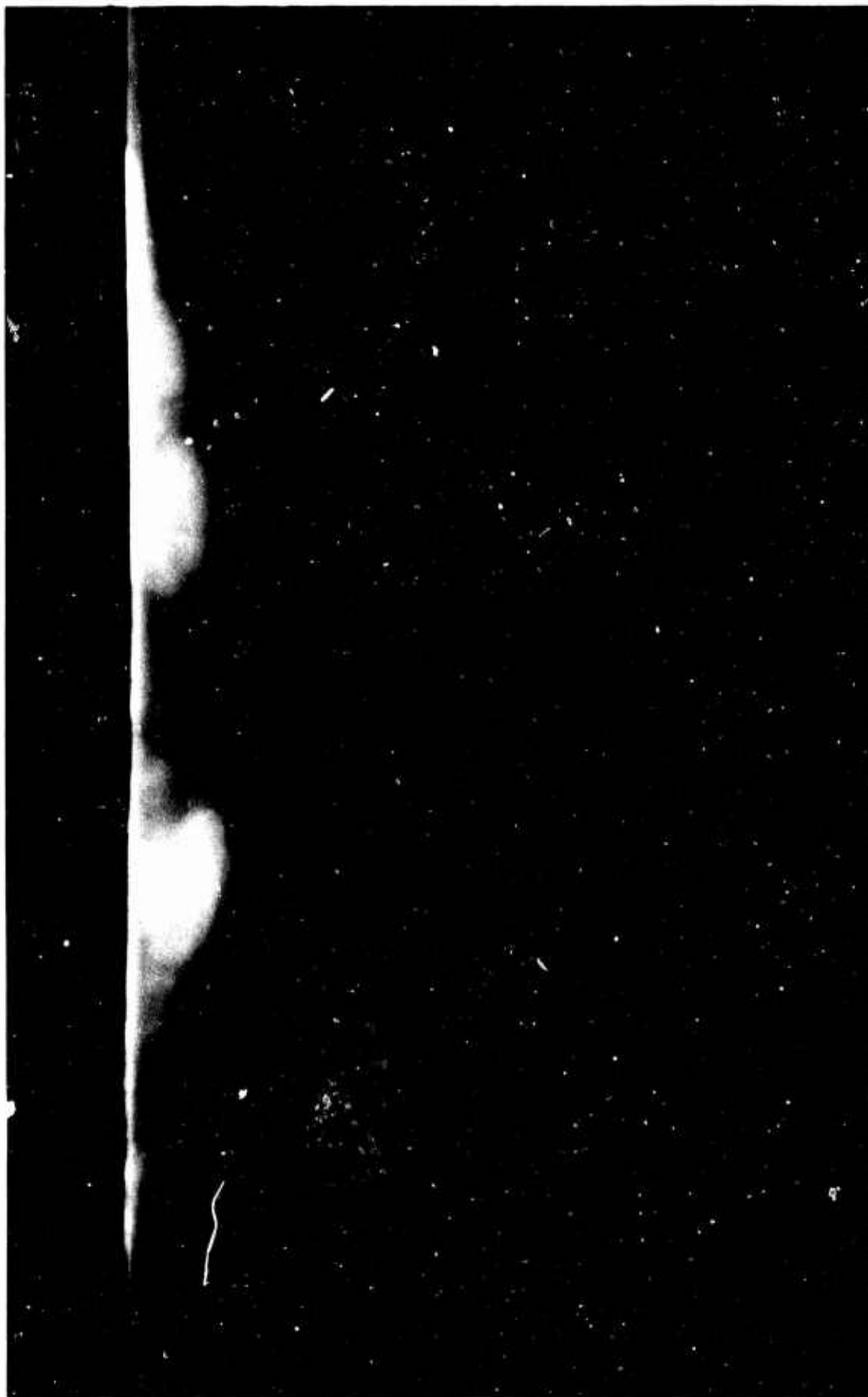
FIGURE 9.   RADIOGRAPH OF THE TOP PORTION OF 50-POUND INGOT (HEAT F) OF LA141 ALLOY

Shrinkage voids were confined to the top 1-1/2 inches of ingot.

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PERFORATED STIRRING
AND SKIMMING DISC

a. ORIGINAL DESIGN

b. MODIFIED DESIGN

HINGED DISC
READY FOR
INSERTING
INTO THE MELT

DROSS
RETAINING
PLATE

DISC INSERTED
FOR STIRRING
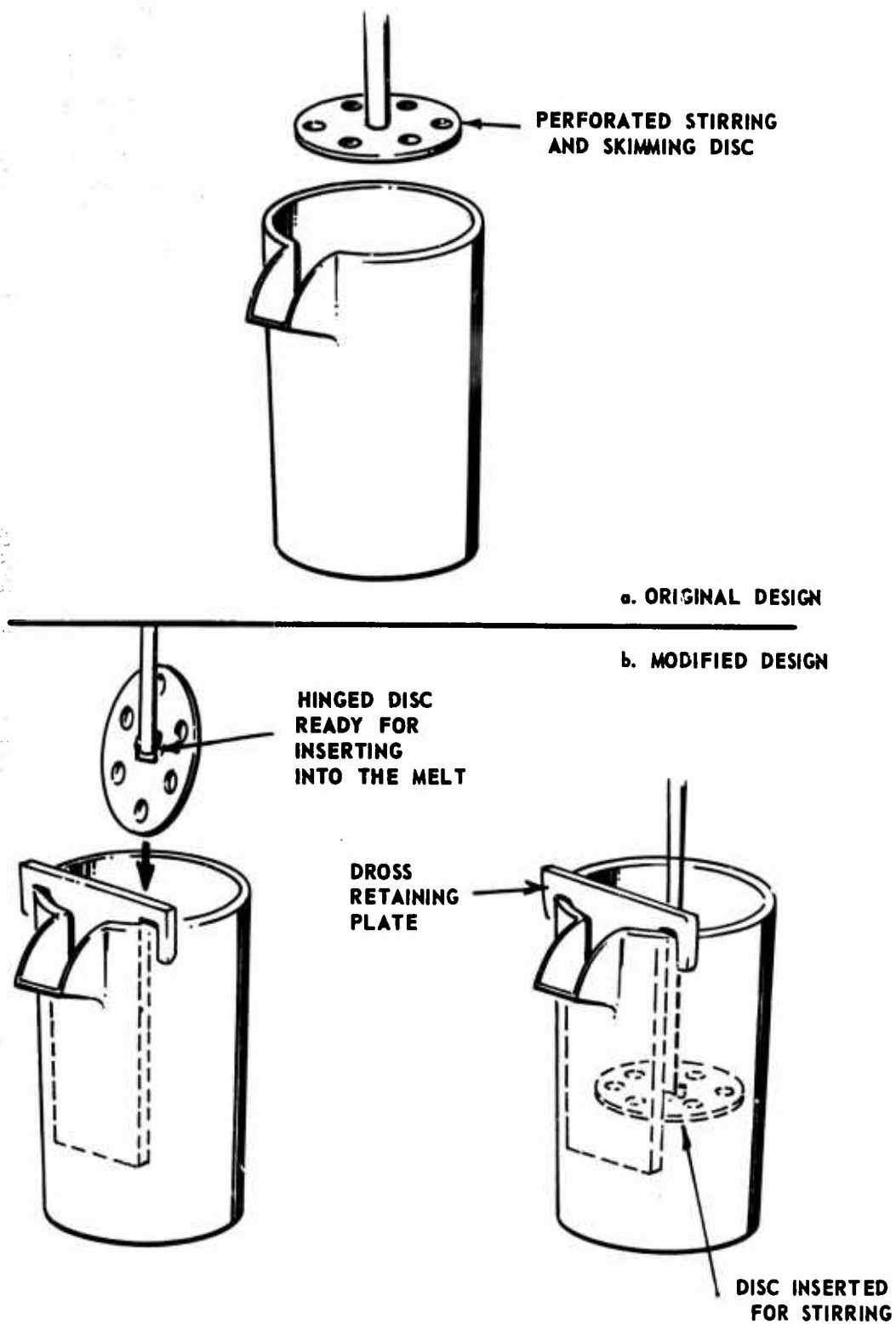
FIGURE 10. EQUIPMENT DESIGNS FOR STIRRING AND SKIMMING
OF MOLTEN LA141 ALLOY HEATS

(1) Charge clean, dry metal to the steel crucible mounted in the induction furnace.

(2) Evacuate chamber to below 10 microns of mercury pressure and continue pumpdown until the leak rate is maintained below 10 microns per minute.

(3) Backfill with argon to a total pressure of 1 inch mercury.

(4) Induction heat the steel crucible to melt the charged material (Melt-down time is about 30-40 minutes.)

(5) Stir melt vigorously, allow 5 minutes for settling, reheat to temperature (1300-1400 F), and pour into tapered (5 degree) steel mold.

TABLE III. PROCESS CONDITIONS FOR MELTING AND CASTING
100-POUND HEATS[a] OF LA141 ALLOY

Heat	Melt Size, lb	Aluminum-Foil Wrapped Lithium	Inert Atmosphere	System Pressure, in of Hg	Pouring Temperature, F	Cast-Ingot Weight, lb
G	100	Yes[b]	Liquid argon[c]	1.0	1312	96
H	100	Yes[b]	Liquid argon	1.0[d]	1350	99

(a) Cost ingot size of approximately 12 x 12 x 14 inches.
(b) Foil wrapped under argon atmosphere.
(c) Moisture content = 2 grains H_2O/1000 cu ft of gas.
(d) Leak rate near end of melting cycle resulted in system pressure of 1.5 inches during the pour.

Heats G and H. In the general scale-up from 50- to 100-pound heats, the only problem of any significance was the inability to measure the melt temperature below a 4-inch depth (total melt depth of ~20 inches) due to the short sheath length on the immersion thermocouple. Under the present equipment setup, a longer thermocouple could not be accommodated. However, the melts were stirred prior to temperature measurement and it is estimated that the melt temperature does not vary by more than 50-75 degrees from top to bottom of the bath.

Figure 11 shows the ingot cast from the first 100-pound heat of LA141 alloy. The radiograph of this ingot as shown in Figure 12 had solidification shrinkage confined to the top 3 inches of the ingot.

The second heat cast (Heat H) was the victim of power-supply difficulties during meltdown and an excessive leak rate which became noticeable after meltdown was completed. As a result of the power difficulties, the total melting and casting cycle was extended by 50 percent over that required for Heat G. The excessive leak rate caused considerable dross formation after meltdown had been realized.

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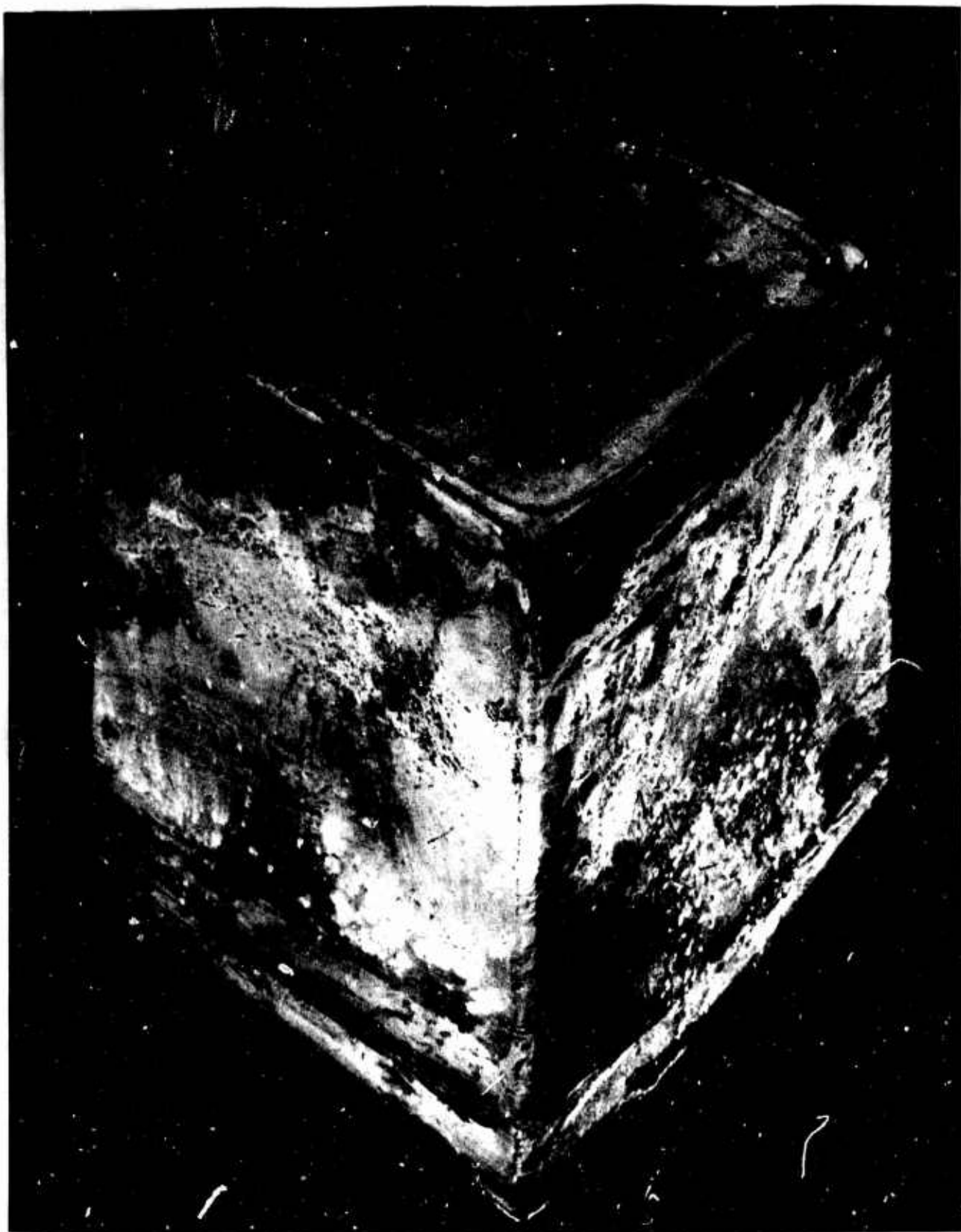
FIGURE 11. 100-POUND LA141 ALLOY INGOT CAST AT BATTELLE

Heat G-ingot size of 12 x 12 x 14 inches.

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FIGURE 12. RADIOGRAPH OF 100-POUND LA141 ALLOY INGOT

Heat G.

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In heating to the pouring temperature after stirring, the melt became overheated and it was decided to pour the ingot at the slightly higher temperature rather than wait for the melt to cool and risk further contamination from the high leak rate.

The resulting ingot was not as clean as Heat G and the surfaces exhibited numerous folds and laps. Radiographs indicated that less than 1/2-inch depth of solidification shrinkage was present in this ingot.

Heat G was processed into plate and sheet for quality-evaluation studies. Approximately 16 pounds of material from this heat and Heat E were sent to Lockheed Missiles and Space Company for use in fabrication studies.

Attempts to process Heat H were not satisfactory as considerable cracking occurred during forging and initial breakdown by hot rolling. Chemical analysis showed considerable variation in lithium content from top to bottom of the ingot (7.2 percent, top; 29.4 percent, bottom) as an apparent result of inadequate stirring of the melt.

Apart from the compositional difficulties, evaluation of the ingot showed it to be sound and of low impurity content as judged by metallographic examination.

Despite operational difficulties with the casting of the second 100-pound ingot, the scale-up of the process to accommodate 100-pound heats was successfully demonstrated. Inasmuch as over 200 pounds of sheet and plate material was available from the material cast previously and process procedures were successfully developed, it was decided to discontinue production of 100-pound heats.

Program Modifications

In view of the success obtained in the casting of 50- and 100-pound heats of LA141 alloy by the vacuum-induction technique, discussions were held between Battelle and the Air Force Materials Laboratory concerning changes in the Phase II program. It was decided that the following changes would be made in the contract requirements:

(1) A minimum of two (instead of six) 100-pound heats of LA141 alloy would be cast

(2) All requirements for the casting of 100-pound heats of LAZ933 alloy would be deleted

The success of the research with 50- and 100-pound heats precluded the need to cast additional heats since process conditions had been optimized within the limitations of existing equipment, and sufficient material was available for evaluation of quality, fabrication studies, mechanical properties, etc. The deletion of LAZ933 from the program was warranted since this alloy has found only very limited use and process parameters established for LA141 would require only minimal modifications to be adaptable to this alloy. These conditions could easily be determined at some future date if markets for this alloy were to develop.

VIII

FABRICATION AND QUALITY EVALUATION STUDIES

The objectives in this portion of the research program were to evaluate (1) the effect of ingot-processing variables on the mechanical properties of LA141 alloy sheet and (2) the quality of the cast ingots being produced. The fabrication of Mg-Li alloys has never been considered a difficult task due to the high degree of workability of this material. Rolling temperatures may vary from 350 to 600 F using pass reductions of 10 to 25 percent.

As a result, rolling procedures have been developed (such as those outlined in the state-of-the-art survey in the Appendix I) without detailed investigations of the effects of process variables on the properties of the sheet. Such documentation could be important in providing a better understanding of some of the fabrication problems being encountered by the users of Mg-Li alloys.

Investigation of Ingot-Processing Parameters

Cast material from Heats C and D were used in evaluating the effects of (1) ingot homogenizing temperature (2) cross rolling during ingot breakdown and (3) rolling-temperature variations on the properties of rolled sheet.

The steeply tapered ingots shown previously in Figure 6 were cropped to provide sound metal and quartered as shown in Figure 13. These quarter-sections were homogenized for times varying from 8 to 48 hours, and forged to rectangular slabs.

The top and bottom surfaces of the forged slabs were scalped to provide good surfaces for subsequent rolling to sheet. Original ingot orientation was identified throughout the various fabrication operations.

All ingot sections were rolled to 1/2-inch-thick plate at a rolling temperature of 500 F. After sectioning, plates were processed to 0.035-inch-thick sheet at rolling temperatures of 350 or 500 F and finally given a 10 percent cold reduction to 0.032 inch to flatten the sheet. Cross rolling was also evaluated during this processing. Figure 14 shows schematically the rolling schedules used. Table IV lists the specific identification of ingot sections and the fabrication conditions of each.

The material cast in Heats E and F were fabricated in a similar manner to provide plate material for ultrasonic evaluation of quality by Brooks and Perkins and IBM's Federal Systems Division. The cast ingots were cropped, sectioned, and homogenized for 20 hours at 500 F. These ingot sections were shown previously in Figure 8.

After forging to rectangular slabs at 500 F, the top and bottom surfaces were milled to remove approximately 1/16 inch of material. These slabs were then rolled to 0.400-inch-thick plate, as shown in Figure 15.

Cropped
ingot

Quartered
ingot

Quarter
section
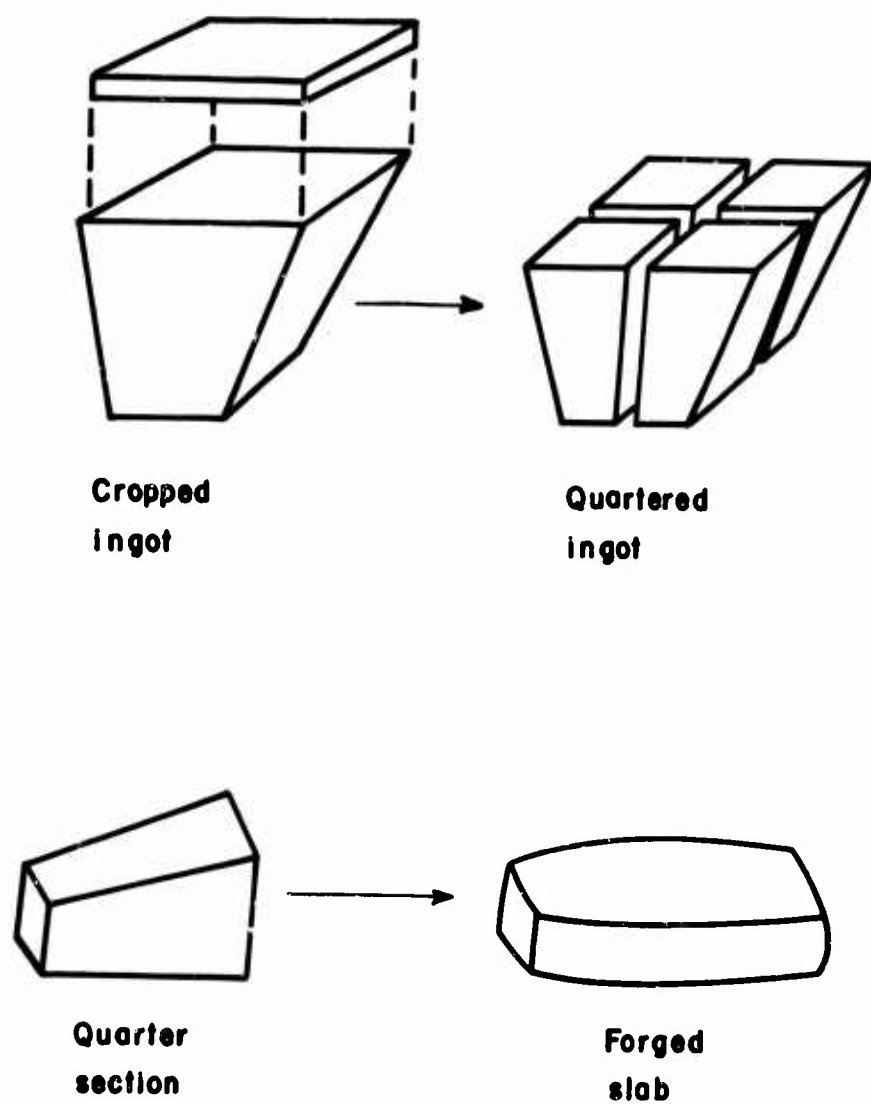
Forged
slab

A55323

FIGURE 13. PROCESSING TECHNIQUES FOR FABRICATION OF FORGED
ROLLING SLABS FROM CAST A141 ALLOY INGOTS

Forged Slabs of LA141 Alloy

Hot Rolled at 500 F
to
1-Inch-Thick Plate

Continued Rolling
at 500 F to ½-Inch-
Thick Plate

Sectioned

Cross Rolled
at 500 F to ½-Inch-
Thick Plate

Sectioned

Continued
Rolling
at 350 F
to 0.035-Inch-
Thick Sheet

Continued
Rolling
at 500 F
to 0.035-Inch-
Thick Sheet

Continued Rolling
at 350 F
in Original Roll Direction
to 0.035-Inch-
Thick Sheet

Continued Rolling
at 500 F
in Original Roll Direction
to 0.035-Inch-
Thick Sheet

Cold Rolled
to
0.032-Inch-
Thick Sheet

A-65340

Evaluation Studies

FIGURE 14. SCHEMATIC DIAGRAM OF SHEET-ROLLING SCHEDULES
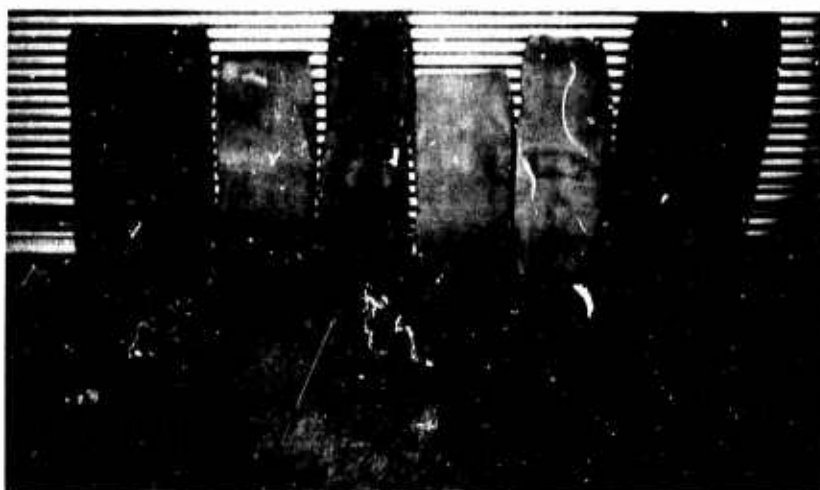
36366

FIGURE 15. AS-ROLLED 0.400-INCH-THICK LA141 ALLOY PLATE

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TABLE IV. PROCESSING CONDITIONS FOR INGOT SECTIONS FROM HEATS C AND D OF LA141 ALLOY

Ingot Section	Code Number	Homogenizing Time, hours at 500 F	Cross Rolled	Hot Rolling[a] Temperature, F
C-1-1	1	8	Yes	350
C-1-2	2	8	Yes	500
C-2-1	3	16	No	350
C-2-2	4	16	No	500
C-3-1	5	28	No	350
C-3-2	6	28	No	500
C-4-1	7	48	Yes	350
C-4-2	8	48	Yes	500
D-1-1	9	8	Yes	350
D-1-2	10	8	Yes	500
D-2-1	11	16	No	350
D-2-2	12	16	No	500
D-3-1	13	28	Yes	350
D-3-2	14	28	Yes	500
D-4-1	15	48	No	350
D-4-2	16	48	No	500

(a) All initial breakdown to 1/2-inch-thick plate was done at 500 F.

Evaluation of Sheet Properties

The effects of processing variables on sheet properties and an evaluation of ingot quality were measured by

(1) Longitudinal and transverse mechanical properties

(2) 45 and 90 degree bend tests

(3) Metallographic examination.

Bend tests were made on both "as-rolled" material and given the standard-T7 heat treatment. Mechanical properties were determined only on material in the -T7 condition.

Mechanical Properties. Mechanical-property determinations were made on both longitudinal and transverse specimens of LA141 alloy sheet in the -T7 condition. Standard 0.505 sheet tensiles were prepared. Tensiles were loaded at a strain rate of 0.02 inch per minute to yield and 0.10 inch per minute to fracture. Six tensiles were tested in each of the 16 processing conditions indicated in Table IV.

Table V lists the average tensile data obtained for the corresponding sheet samples. Figures 16, 17, and 18 show the effects of rolling temperature on sheet properties for ingots having various homogenizing treatments.

TABLE V. EFFECT OF PROCESSING VARIABLES ON THE MECHANICAL PROPERTIES OF 1/32-INCH-THICK LA141 ALLOY SHEET[a]

Code Number	Homogenizing Time, hours at 500 F	Cross Rolled	Hot-Rolling Temperature, F	Tensile-Test Direction	Mechanical Properties[b]		
					Ultimate Tensile Strength, 1000 psi	Yield Strength, 1000 psi	Elongation, percent in 2 inches
1	8	Yes	350	L	21.1	18.3	26.0
		Yes		T	23.7	20.3	13.0
9	8	Yes	350	L	21.0	18.7	28.5
		Yes		T	23.5	20.9	16.0
2	8	Yes	500	L	22.2	20.3	27.5
		Yes		T	23.5	21.7	19.0
10	8	Yes	500	L	23.2	21.6	23.0
		Yes		T	24.1	22.6	18.0
3	16	No	350	L	20.4	16.9	25.0
		No		T	22.3	18.4	20.5
11	16	No	350	L	20.1	16.8	30.5
		No		T	22.1	18.6	21.0
4	16	No	500	L	22.7	21.1	21.0
		No		T	23.9	22.3	15.0
12	16	No	500	L	22.5	20.9	25.0
		No		T	23.3	21.9	19.5
5	28	No	350	L	20.3	16.8	26.5
		No		T	22.2	18.4	23.0
13	28	Yes	350	L	20.7	18.7	27.8
		Yes		T	23.5	21.0	17.5
6	28	No	500	L	23.4	21.6	22.5
		No		T	24.3	22.9	18.0
14	28	Yes	500	L	23.0	21.3	22.5
		Yes		T	24.4	22.8	19.0
7	48	Yes	350	L	21.3	18.9	32.0
		Yes		T	23.8	20.9	22.6
15	48	No	350	L	20.5	17.8	31.5
		No		T	22.5	19.7	21.5
8	48	Yes	500	L	23.3	21.6	22.0
		Yes		T	24.4	22.9	19.5
		No	500	L	22.6	20.8	25.0
		No		T	23.8	22.4	17.5

(a) All sheet material heat treated 6 hrs. at 350 F prior to testing.
(b) Data represents the average of 6 tensile tests in each condition.

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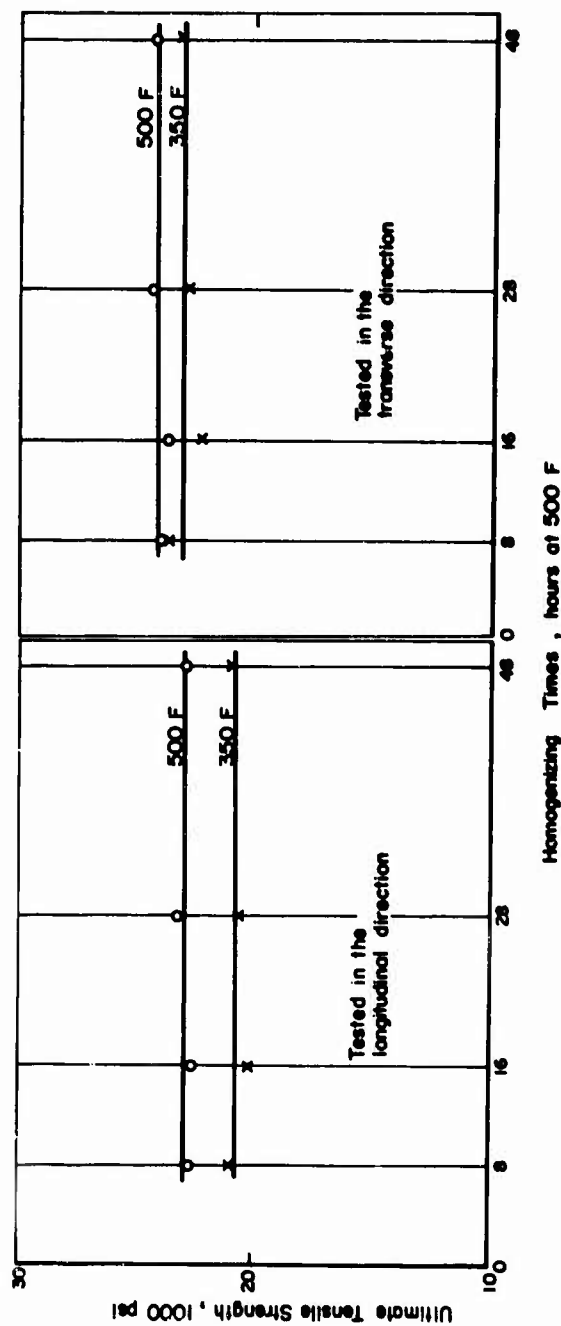
FIGURE 16. EFFECT OF ROLLING TEMPERATURE AND INGOT HOMOGENIZING TIMES ON ULTIMATE TENSILE STRENGTH OF 1/32-INCH-THICK LA141 ALLOY SHEET

Sheet material heat treated 6 hours at 350 F prior to testing.
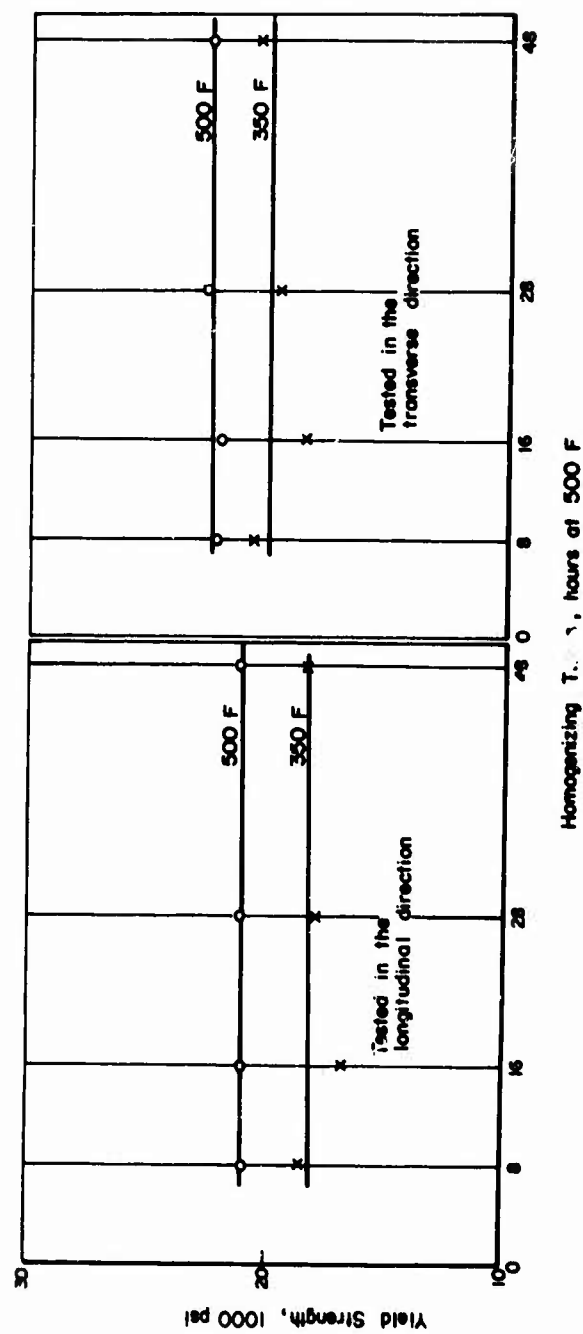
FIGURE 17. EFFECT OF ROLLING TEMPERATURE AND INGOT HOMOGENIZING TIMES ON YIELD STRENGTH OF 1/32-INCH-THICK LA141 ALLOY SHEET

Sheet material heat treated 6 hours at 350 F prior to testing.
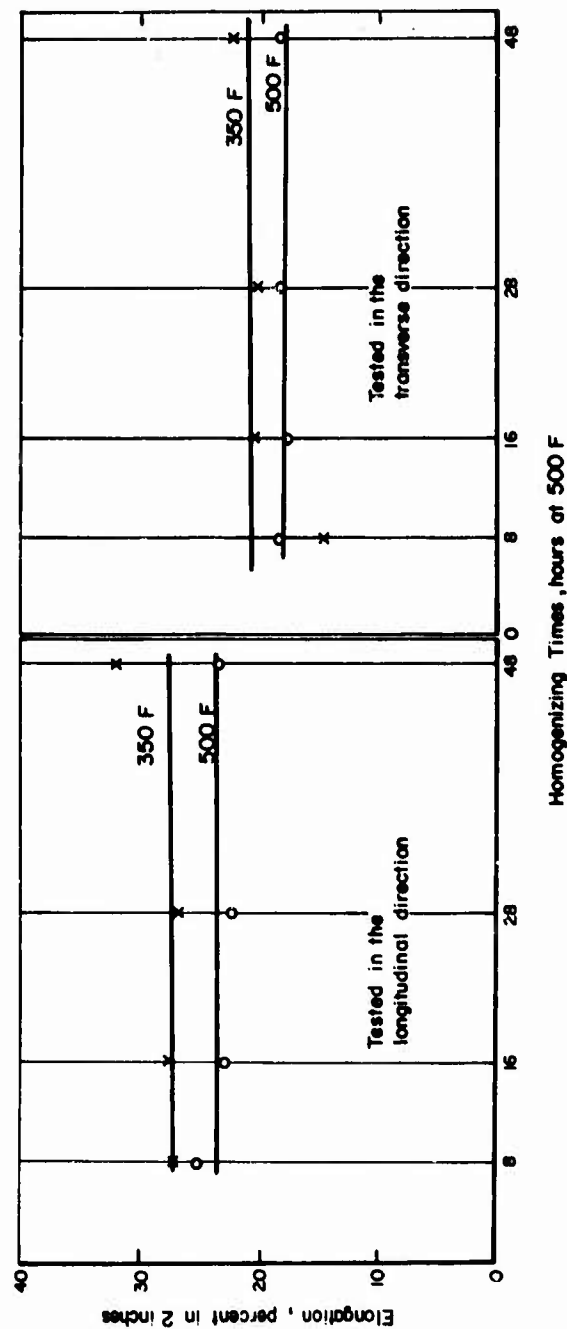
FIGURE 18. EFFECT OF ROLLING TEMPERATURE AND INGOT HOMOGENIZING TIMES ON TENSILE ELONGATION OF 1/32-INCH-THICK LA141 ALLOY SHEET   A-55320

Sheet materials heat treated 6 hours at 350 F prior to testing.

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The results show that:

(1) Cross-rolling effects on mechanical properties could not be detected.

(2) Ultimate and yield strengths were approximately 15 percent higher for material rolled at 500 F compared to those of sheet rolled at 350 F.

(3) Tensile elongations showed a comparable decrease, although results showed more variation than were encountered in strength properties. (Metallographic examinations failed to explain these variations.)

These mechanical-property data show slightly higher strength properties with equivalent elongations when compared to typical properties reported in previous work by Battelle, Lockheed, and others. These improvements presumably reflect a higher quality material.

Bend Tests. Bend tests were made on 1/32-inch-thick sheet at both 90 and 45 degrees to the rolling direction. Six tests were made for each of the 16 sheet conditions listed previously in Table IV. Samples were evaluated on their ability to withstand a 90-degree bend over a 1T radius.

Data on as-rolled material is listed in Table VI along with data on material which was heat treated at 350 F for 6 hours. The data show that:

(1) Variations in sheet processing did not effect 1T bend ductility on material in the stabilized condition.

(2) Material rolled at 350 F withstood a 1T bend in the as-rolled condition while material rolled at 500 F did not.

The good bend-test results on material in the -T7 condition are typical of 1T bend data on LA141 alloy. The differences shown in the bend tests on as-rolled sheet reflect the rolling-temperature variations as indicated above. In the sheet rolled at 500 F, the Al-Li precipitate is still in solution or is just beginning to precipitate at the grain boundaries of an equiaxed grain structure. Therefore, the bend ductility is not optimum. The material rolled at 350 F is finer grained by virtue of its worked structure and also softer since it is overaged with respect to the Al-Li precipitation. Metallographic examination and hardness data confirmed these differences.

Cross-rolling variations and differences in homogenizing times were not reflected in the bend-test data.

TABLE VI. EFFECT OF SHEET PROCESSING PARAMETERS ON IT BEND DUCTILITY OF
1/32-INCH-THICK LA141 ALLOY SHEET

Ingot	Code Number	Ingot Homogenizing Time, hours	Cross Rolled During Processing	Hot-Rolling Temperature, F	Results[a] of 90-Degree Bends Over 1T Die			
					As-Rolled Condition		-T7 Condition[b]	
					90 Degree to Rolling Direction	45 Degree to Rolling Direction	90 Degree to Rolling Direction	45 Degree to Rolling Direction
C	1	8	Yes	350	Good	Good	Good	Good
D	9	8	Yes	350	Good	Good/Cracked	Good	Good
C	2	8	Yes	500	Fracture	Fracture	Good	Good
D	10	8	Yes	500	Fracture	Fracture	Good	Good
C	3	16	No	350	Good	Cracked	Good	Good
D	11	16	No	350	Good	Good	Good	Good
C	4	16	No	500	Fracture	Fracture	Good	Good
D	12	16	No	500	Fracture	Fracture	Good	Good
C	5	28	No	350	Good	Cracked	Good	Good
D	13	28	Yes	350	Good	Good	Good	Good
C	6	28	No	500	Fracture	Fracture	Good	Good
D	14	28	Yes	500	Fracture	Fracture	Good	Good
C	7	48	Yes	350	Good	Good/Cracked	Good	Good
D	15	48	No	350	Good	Good	Good	Good
C	8	48	Yes	500	Fracture	Fracture	Good	Good
D	16	48	No	500	Fracture	Fracture	Good	Good

(a) Six bend tests made for each sheet condition.   (b) Held at 350 F for 6 hours, air cooled.

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Metallographic Examination. A representative microstructure of LA141 alloy sheet
is shown in Figure 19. Material processed from the 50-pound ingots showed generally
clean microstructures although some dross entrapment was noted in a few areas.
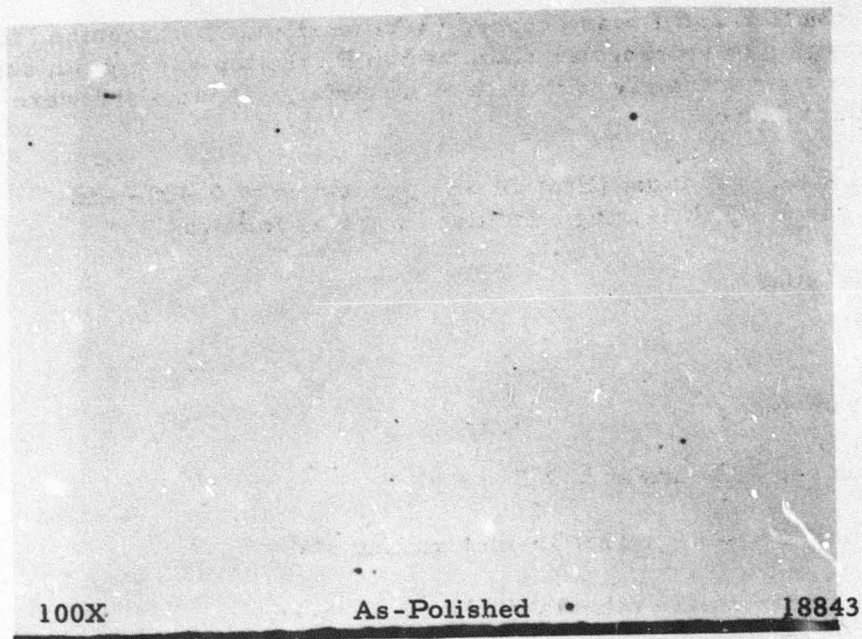
FIGURE 19. MICROSTRUC-
TURE OF SHEET MATERIAL
FROM 50-POUND HEATS OF
LA141 ALLOY

100X   As-Polished   18843

Conclusions

These studies show that the LA141 alloy can be easily fabricated over a range of
processing conditions if the starting material is of high quality. Rolling at temperatures
at or near 500 F has been used previously and these studies indicate this to be probably
an optimum temperature for hot rolling, provided the material is subsequently stabilized
at a lower temperature. Effects of processing variables are essentially eliminated by
rolling at this temperature, and finished sheet has good ductility.

Quality Evaluations

Extensive examination of ingot quality was conducted on two 50-pound ingots (Heats E and F) and on one 100-pound ingot (Heat G).

The material cast in Heats E and F was cropped, sectioned, and homogenized for 20 hours at 500 F. After forging to rectangular slabs at 500 F, the top and bottom surfaces were milled to remove approximately 1/16 inch of material. These slabs were then rolled to 0.400-inch-thick plate.

The first 100-pound LA141 cast ingot (Heat G) was processed to 0.400- and 0 065-inch-thick plate and sheet. Fabricating conditions were as follows:

(1) Preparation for forging

      (a) crop
      (b) cut in half
      (c) machine all surfaces

(2) Homogenizing soak for 20 hours at 500 F

(3) Press forge to fabricate two 4 x 11 x 15-inch rolling slabs

(4) Roll at 500 F with intermediate reheats to plate and sheet.

A yield of ~71 percent was realized on this first 100-pound heat as shown below:

Melt weight		100 lb
Cast weight		96 lb
After cropping		70. 5 lb
After cutting and	1 -	34. 5 lb
machining	1 -	33. 25 lb
		67. 75 lb yield in plate
		and sheet

$$\frac{67.75}{96} \times 100 = 70.6 \text{ percent yield.}$$

As the figures show, most of the weight loss (27 percent) was in the cropped end. No attempt was made in these studies to develop techniques for minimizing or completely eliminating shrinkage pipe in the cast ingot. Thus, ingot yields in this program were always below what could be realized in a commercial-size operation where different mold designs and ingot-cooling techniques could be used to reduce shrink-cavity losses.

Evaluation of Sheet Properties

The ingot quality of Heats E, F, and G was evaluated by:

(1) Longitudinal and transverse mechanical properties

(2) 90 degree bend tests

(3) Metallographic examination

(4) Ultrasonic inspection

(5) General quality inspection and evaluation by IBM and Lockheed.

Heats E and F. Six plates totalling 60 pounds of 0.400-inch-thick rolled material were transported to Brooks and Perkins for ultrasonic inspection. The results indicated that:

(1) All material met or surpassed the Class C (8/64 inch max. defect) inspection classification now considered acceptable for commercial products.

(2) Two plates had Class C defects — one in each plate near the plate edge.

(3) One plate had two Class B defects (5/64 inch max.) and seven Class A defects (3/64 inch max.).

(4) Three plates showed no defects.

A total of 28 pounds of this same material was subsequently sent to IBM-Federal Systems Division for examination. Their investigations were quite extensive and their report to Battelle dated April 24, 1967, concluded that:

1. The plate is of high quality with mechanical properties and microstructural cleanliness superior to existing commercial grade stock. It ranks as the the best plate stock ever observed at IBM Owego.

2. Ultrasonically the material was relatively free of defects in excess of 5/64-inch diameter, thereby meeting a grade B or No. 5 acceptance standard. This is one grade higher in quality than that now commercially available to IBM.

No further mechanical-property testing or quality evaluations other than metallographic examination were performed on this material at Battelle. Metallographic examinations showed that material from these heats was of good quality and showed slightly better internal cleanliness than those of Heats C and D.

Heat G. Mechanical-property determinations and bend tests were made on representative samples of 1/16-inch-thick sheet in both the longitudinal and transverse directions after rolling at 500 F and the commercial -T7 heat treatment. The results are listed in Table VII.

TABLE VII. MECHANICAL-PROPERTY DATA ON 0.063-INCH-THICK SHEET FABRICATED FROM A 100-POUND VACUUM-INDUCTION-MELTED INGOT OF LA141 ALLOY (HEAT G)

Number of Tests	Specimen Orientation	Mechanical Properties[a]			
		Ultimate Tensile Strength, 1,000 psi	Yield Tensile Strength, 1,000 psi	Elongation, percent in 2 inches	Summary of Transverse 1T Bend Tests
8	L	19.7-19.9	14.5-15.0	40-45	OK
8	T	20.2-20.5	15.1-16.0	35-47	OK

(a) -T7 Condition.

Standard V-notch Charpy impact tests were also made on this material using 2/3-size Charpy impact specimens. Results on material tested at room temperature after heat treatment at 350 F for 6 hours are:

Specimen Orientation	Impact Energy, ft-lb
L	8.5
T	13-14

These data compare favorably with information supplied by Lockheed in previous property evaluation studies on LA141 alloy.

Metallographic examination showed generally clean microstructures, although some dross entrapment is still noted in a few areas. Figure 20 shows a sample of as-rolled material after a re-solution heat treatment of 1/2 hour at 500 F.
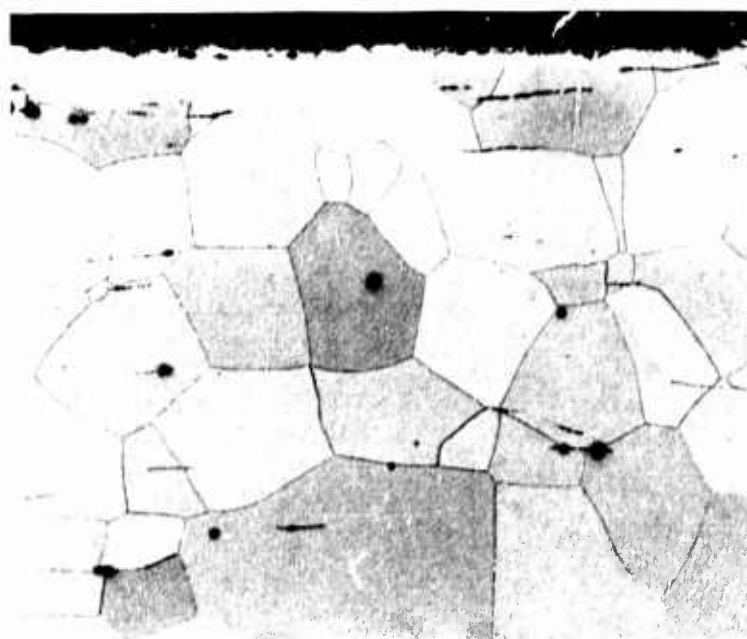
250X   8B434

FIGURE 20. MICROSTRUCTURE OF RECRYSTALLIZED
          SHEET MATERIAL FROM 100-POUND
          HEAT OF LA141 ALLOY

EFFECT OF SURFACE CONDITION
ON PROPERTIES OF ROLLED SHEET

It was determined in earlier studies[5] on Mg-Li alloys that lithium appears to be depleted from the surface of certain Mg-Li alloys (particularly those having an "$\alpha + \beta$" structure) at elevated temperatures in a vacuum. It was not known, however, how extensive this depletion was or what effects this lithium loss might have on properties or fabricability of LA141 sheet. Because of the known problems in fabrication of commercial LA141 alloy, an investigation of lithium depletion, its extent, and its effect on mechanical properties was conducted as a part of Phase I.

Samples of LA141 alloy measuring 0.25 x 2 x 2 inches were machined to size, cleaned, and weighed. One group of samples was coated with sodium silicate solution. A second group was coated with Dow 997 silicone lacquer. These samples, along with uncoated specimens, underwent a time-temperature exposure sequence in air. Data are recorded in Table VII along with the weight change of samples before and after treatment. The erratic weight changes gave no concrete information as to lithium depletion. The "before" and "after" samples are shown in Figure 21.

TABLE VIII. EFFECT OF TIME AT ELEVATED TEMPERATURES ON WEIGHT
CHANGES OF LA141 ALLOY SHEET SAMPLES

Exposure Time, hours	Sample Condition	Sample Weight Change in Milligrams at Indicated Exposure Temperature, F	
		400	600
2	Uncoated Sample	0.000	-0.033
	Coated with Na_2SiO_4	+0.001	-0.058[a]
	Coated with Dow 997	-0.012	0.000
4	Uncoated Sample	-0.002	-0.005
	Coated with Na_2SiO_4	-0.005	-0.182[a]
	Coated with Dow 997	-0.029	-0.017
16	Uncoated Sample	-0.001	-0.001
	Coated with Na_2SiO_4	-0.005	-0.616[a]
	Coated with Dow 997	-0.018	-0.004

(a) Alloy was attacked by Na_2SiO_4 at high temperatures.

Samples were sectioned and their microstructures examined. No visible indication of surface lithium depletion could be detected. Since only the beta phase exists in the solid solution of this alloy, it is possible that lithium loss could have occurred but was not detected because the lithium level was still above the 11 percent that divides "β" and "$\alpha + \beta$" phase fields.

Chemical analyses were checked on an as-rolled sample and on one uncoated sample that was heated in air for 16 hours at 600 F. Layers 0.002 inch thick were machined from the surface of the 1/4-inch-thick samples. Samples were also taken 1/16 inch below the surface and 1/8 inch below the surface (or in the center of the sample). The results given in Table IX show that (1) before treatment the as rolled sample already had some surface lithium depletion and (2) heating for 16 hours at 600 F increased the lithium depletion on the surface only slightly

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Sample Before Treatment   Uncoated 16 hours at 600 F
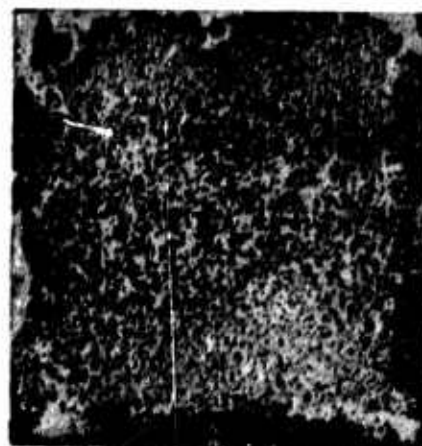
Dow "997" Silicone Lacquer   Sodium Silicate 16 hours at
16 hours at 600 F   600 F

35811

FIGURE 21. APPEARANCE OF COATED AND UNCOATED SHEET SAMPLES
AFTER EXPOSURE TO AIR FOR TIMES AND TEMPERATURES
INDICATED

TABLE IX EFFECT OF ELEVATED-TEMPERATURE EXPOSURE ON LITHIUM
DEPLETION OF LA141 ALLOY PLATE SURFACES

Condition	Sample Location Depth Below Surface, inch	Lithium Content, percent
As-Rolled Plate, rolled at 500 F	0.002	12.4
	1/16	13.1
	1/8	13.2
As-Rolled Plate, reheated for 16 hours at 600 F	0.002	11.6
	1/16	13.2
	1/8	13.1

These results indicated that surface depletion of lithium during heating was not sufficient to cause problems in subsequent fabrication operations. However, mechanical-property evaluations were made on sheet samples exposed to elevated temperatures for an extended period to clarify further the effect of surface characteristics on fabricability and material properties.

Standard size 1/32-inch-thick sheet tensile specimens were given a 42-hour exposure to air at 500 F followed by 3-6 hours at 350 F, and air cooled. The usual procedure for cleaning tensile specimens prior to testing is "scrubbing" with fine steel wool and water. It was decided to clean some additional specimens with an acid pickle and with a light grit blast to evaluate surface effects on the property determinations of this thin sheet material.

The results of these studies, as shown in Table X, indicate no significant change in tensile strengths regardless of surface treatment. The elongation values were reduced, however, by one-third with long-time exposure at 500 F and even more when specimens were pickled or grit blasted.

The ductility of these thin-sheet specimens is obviously reduced by long-time elevated-temperature exposure. However, these test conditions were exaggerated well beyond the normal exposure times required for sheet fabrication. Also, any surface effects from the long-time exposure at 500 F or the cleaning procedures used would be more pronounced on the thin-sheet cross section used in these particular studies.

Thus, the sheet properties determined in the studies shown here do not suggest any problems in normal processing of Mg-Li alloys and preclude the need for protective coatings during fabrication.

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TABLE X. EFFECT OF ELEVATED-TEMPERATURE EXPOSURE AND
SUBSEQUENT SURFACE TREATMENT ON MECHANICAL
PROPERTIES AND BEND DUCTILITY OF 1/32-INCH-
THICK LA141 ALLOY SHEET

| Exposure Cycle | Surface Treatment Prior to Testing | Mechanical Properties | | | |
		Ultimate Tensile Strength, 1000 psi	Yield Tensile Strength, 1000 psi	Elongation, percent in 2 inches	Results of 1T Bend Tests
1 hour at 500 F plus 6 hours at 350 F	None	21.5	19.6	27.5	OK
42 hours at 500 F plus 6 hours at 350 F	None	23.9	21.1	18.0	Fractured
42 hours at 500 F plus 6 hours at 350 F	Steel wool/water	22.7	20.2	19.5	Fractured
42 hours at 500 F plus 6 hours at 350 F	H_2SO_4 pickle	23.7	21.2	16.0	Fractured
42 hours at 500 F plus 6 hours at 350 F	Grit blast	23.8	19.8	14.0	Fractured

X

A STUDY OF STABILIZATION TREATMENTS

In all previous Battelle studies involving the LA141 alloy, fabrication at temperatures ranging from 400 to 600 F was followed by aging treatments of 150 to 250 F for time increments to 2000 hours. The objective of the aging treatments is to fully transform the unstable theta phase, $MgLi_2Al$, to the stable phases $MgLi + AlLi$. Figure 22 shows the effect of aluminum content and aging time at 200 F on the hardness of 6Mg/Li-Al alloys. [7] The presence of the theta phase in the alloy system is evident in the hardness curves above 0.5 percent aluminum. In the original development work for NASA on the LA141 alloy, stabilizing treatments were examined and showed that an aging treatment of 24 to 48 hours at 200 F was adequate to stabilize the alloy as shown in Table XI.

There is no documentation available, however, to indicate the source of the 6-hour stabilizing treatment at 350 F which is called for in AMS Specification 4386 for the LA141 alloy in the -T7 condition. This treatment has been used without significant difficulty for some time by Brooks and Perkins on all wrought LA141 alloy products. As a part of the present program, a study of stabilizing treatments has been conducted to examine the effects of the "350 F-6 hour" treatment as well as other aging temperatures and times.

Of particular interest along similar lines is information received from Lockheed Missiles and Space Company on studies conducted at both their Palo Alto Research Laboratory and Sunnyvale facilities. The Lockheed studies were prompted by the problems encountered with low-ductility LA141 sheet over the past year — the same problems which in part prompted this current program at Battelle. No results of the Lockheed studies will be included here since this information has not been published as yet. However, some of these results appear to be in close agreement with information obtained concurrently on this program.

Hardness Survey

Sheet samples from Heat G were solution treated at 500 F for 1 hour, and cold-water quenched prior to reheating at 250, 300, and 350 F for 6 hours and again water quenching. It was anticipated that, if these treatments were truly stabilizing, little or no change should occur in hardness after long-time aging at room temperature.

The curves shown in Figure 23 indicate that for all three temperatures, considerable hardening occurred during room-temperature aging. In fact, only after 336 hours at room temperature does some leveling off of hardness occur in specimens that were aged at 250 to 350 F. Hardnesses have increased by as much as 20 points between aging periods of 24 and 336 hours. Material quenched from 500 F continued to show increasing hardness even after 672 hours at room temperature.

These hardness surveys show that some solution of AlLi does apparently occur during heating at these low temperatures, with theta phase being precipitated during room-temperature aging. However, the cold-water quenches used here are not truly representative of actual process conditions where slower cooling in air is the most common practice both after high-temperature rolling and after the 350 F treatment.

52
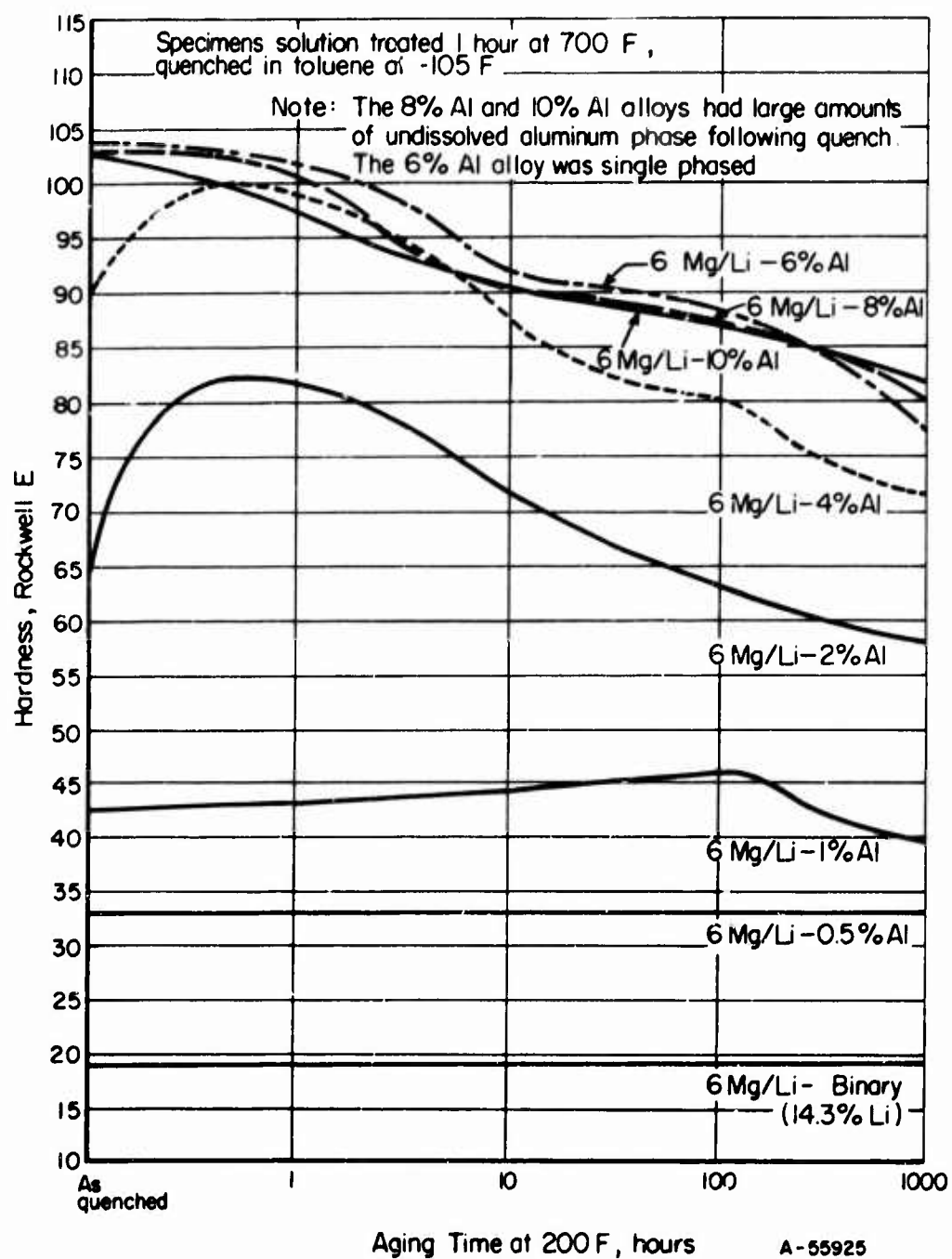
FIGURE 22. EFFECT OF ALUMINUM CONTENT AND AGING TIME AT 200 F
ON HARDNESS LEVEL OF Mg-Li-Al ALLOYS HAVING THE
SAME RATIOS OF MAGNESIUM-TO-LITHIUM CONTENTS[7]

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TABLE XI. MECHANICAL-PROPERTY STABILITY OF Mg-14.1Li-1.5Al[a] ALLOY AFTER EXPOSURE AT 200 F, ROOM TEMPERATURE, AND -100 F[5]

Tested at Room Temperature After Exposures Indicated

Exposure Time, hr	200 F				Room Temperature				-100 F[b]			
	Ultimate Tensile Strength, psi	Yield Tensile Strength, psi	Elongation in 2 In.[c], percent	Hardness, R_E	Ultimate Tensile Strength, psi	Yield Tensile Strength, psi	Elongation in 2 In., percent	Hardness, R_E	Ultimate Tensile Strength, psi	Yield Tensile Strength, psi	Elongation in 2 In., percent	Hardness, R_E
0	22,800 / 23,200	19,500 / 19,700	12.0 / 14.0	58								
4	21,500 / 22,000	18,400 / 19,300	15.5 / 17.0	54								
15	21,500 / 21,600	18,400 / 18,900	18.0 / 22.5	55								
25	21,600 / 21,900	18,500 / 18,800	19.5 / 21.5	56	28,100 / 27,300	26,300 / 26,200	8.0 / 9.0	75				
50	20,700 / 20,800	17,300 / 17,500	23.5 / 21.0	54	29,600 / 29,900	27,900 / 28,300	5.5 / 6.0	76				
100	19,900 / 19,900	16,700 / 17,000	22.5 / 24.0	53	32,500 / 32,300	30,900 / 30,600	2.5 / 3.0	79	24,800 / 25,200	23,000 / 23,700	11.5 / 13.0	61
500	19,200 / 19,500	16,200 / 16,600	25.0 / 25.0	51	34,100 / 34,700	32,500 / 33,000	3.0 / 3.5	80	24,200 / 24,900	22,000 / 23,100	14.0 / 12.0	61
1000	19,000 / 19,100	15,700 / 15,800	23.5 / 20.5	50	33,300 / 33,400	31,300 / 31,600	4.5 / 4.0	79	25,200 / 25,600	23,500 / 23,700	13.0 / 10.5	62
1500	18,400 / 18,900	15,000 / 15,800	32.0 / 26.0	49	32,000 / 31,600	30,000 / 29,800	6.0 / 8.5	77	25,000 / 25,600	23,200 / 23,600	11.5 / 12.5	61
2000	18,200 / 18,600	14,900 / 15,300	30.0 / 26.0	45	30,900 / 29,700	28,500 / 27,700	8.0 / 8.0	74	25,700 / 25,800	23,900 / 24,100	9.0 / 9.0	59

(a) Alloy melted as 10-pound heat under 3:1 LiCl:LiF + LiBr flux.
(b) Specimens maintained at -100 F by immersion under crushed Dry Ice.
(c) Transverse tensile specimens: reduced section 2 inches long x 0.5 inch wide x 0.063 inch thick.

54
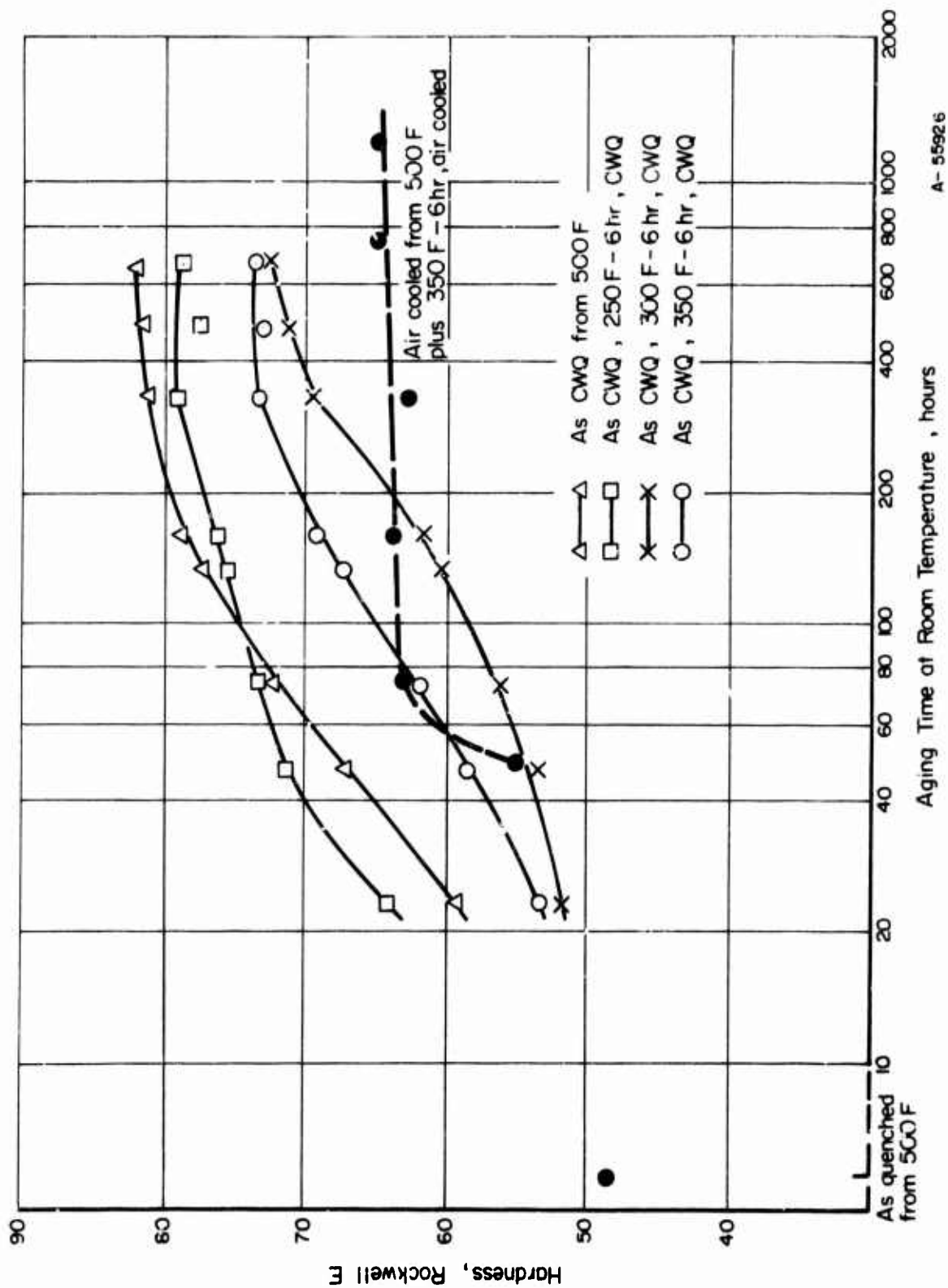
FIGURE 23. EFFECT OF ROOM-TEMPERATURE AGING ON HARDNESS OF HEAT-TREATED LA141 ALLOY SHEET

55

Air cooling had a significant effect as shown also in Figure 23. After the 350 F-6 hour treatment followed by cooling in air, hardnesses increased to a lesser degree and were fully stabilized after 72 hours at room temperature.

Mechanical Properties

Hardness data indicate that significant changes occur while material is aged at room temperature following the 250-350 F treatments. To establish the aging effects on properties more definitely, samples from seven flux-melted scrap heats were used for mechanical-property evaluations of material immediately after the 350 F treatment, and after 168 and 1056 hours at room temperature. The sheet rolled from flux-melted heats was used since it was felt that perhaps less pure material than the vacuum-induction-melted heats might show a greater effect from room-temperature aging due to higher impurity content.

The results shown in Figure 24 substantiate the original findings in the hardness survey. Strength properties show some increase with corresponding decreases in elongation values. However, in no case was ductility sufficiently reduced by room-temperature aging to cause any embrittlement or lack of formability (T bend tests confirmed this). In longitudinal test specimens, elongation values were all above 25 percent even after the 1000-hour room-temperature aging period. The variations in properties from heat to heat reflect variations in Li and Al contents since these were not controlled in the scrap-melting studies.

Charpy impact values also show a change during room-temperature aging following the -T7 heat treatment as indicated in Table XII.

TABLE XII. CHARPY IMPACT DATA ON LA141
ALLOY PLATE[1] IN THE -T7
CONDITION AFTER AGING AT
ROOM TEMPERATURE

Aging Time at Room Temperature Following -T7 Treatment, hours	Impact Energy ft-lb	
	Longitudinal	Transverse
0	13.0/14.0	8.5/8.5
168	10.5/10.5	6.5/6.5
768	9.5/10.5	6.0/6.5

(1) Heat G material.

56
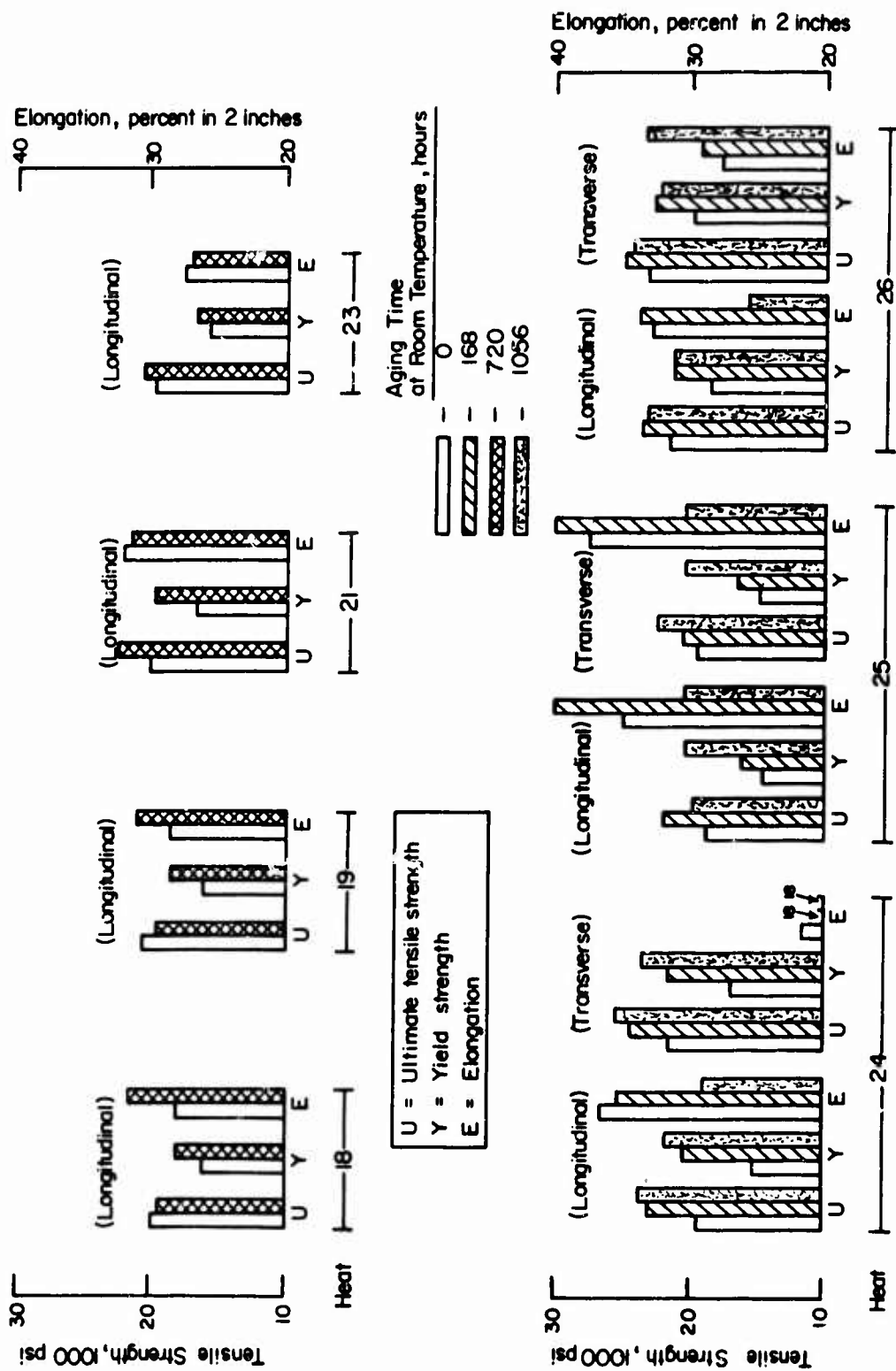
FIGURE 24. EFFECT OF LONG-TIME ROOM-TEMPERATURE AGING ON MECHANICAL PROPERTIES OF HEAT-TREATED LA141 ALLOY SHEET

Sheet rolled at 500 F, air cooled, reheated for 6 hours at 350 F, air cooled.

A—55927

57

As a part of the stabilization study, a hardness survey was made on material rolled at 350 F during the Phase I program. Samples were given the -T7 heat treatment, air cooled, and subsequently aged for 3, 6, and 16 hours at 250 F and 300 F. Results tabulated in Table XIII show that this material was in the fully stabilized condition after the -T7 treatment since subsequent aging treatments were ineffective.

The data would suggest that, if fabrication could be done at temperatures lower than those currently being used, much of the stabilization problem in present-day commercial sheet products might be eliminated. However, the increased rolling loads indigenous to the use of lower rolling temperatures would require the use of stiffer rolling mills. In addition, these lower temperatures could cause more cracking during initial ingot breakdown than is now encountered, with less chance to weld up the cracks as rolling proceeds.

TABLE XIII. EFFECT OF AGING TREATMENTS ON
THE HARDNESS OF LA141 SHEET
ROLLED AT 350 F AND HEAT
TREATED TO THE -T7 CONDITION

| Aging Treatment | | Hardness, |
Time, hours	Temperature, F	Rockwell E
None	--	56
3	250	48
3	300	55
6	250	53
6	300	52
16	250	51
16	300	52

Effects of Grain Size on Ductility After -T7 Heat Treatment

The use of Charpy impact tests to evaluate cast ingot quality is described elsewhere in this report (See Table XIX). Cast material for these tests was homogenized and given the -T7 heat treatment prior to determining impact strengths. A duplicate set of samples was given identical heat treatment and allowed to room-temperature age for 48 days (1152 hours) prior to testing.

Table XIV shows the pronounced decrease in ductility that occurred upon room temperature aging. Figure 25 shows the marked change in appearance of the fractured specimen surfaces after long-time aging has occurred.

These data serve only to point up the actual instability of the -T7 treatment since differences reported in wrought materials are much less pronounced. However, more marked effects of the -T7 treatment could be noted even in wrought material if grain size control was not maintained or if material experienced insufficient working to break up large cast grains.

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TABLE XIV. EFFECT OF ROOM-TEMPERATURE
AGING ON CHARPY IMPACT TEST
VALUES OF CAST LA141 ALLOY
MATERIAL AFTER -T7 HEAT
TREATMENT[a]

Heat	Time at Room Temperature After -T7 Heat Treatment, days	Impact Energy, ft-lb
21	<1	34.0/39.5
	48	9.0/9.0
23	< 1	17.0/17.0
	48	12.0/13.5
24	<1	21.0/24.0
	48	6.5/7.0
25	<1	29.5/36.0
	48	10.0/10.0
26	<1	32.0/30.5
	48	9.0/10.0
G	<1	35.0/32.0
	48	5.0/5.0

(a) 350 F, 6 hours, air cooled

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1C440

a. Room-Temperature Aged < 1 Day Following
   -T7 Heat Treatment

   (Impact energy = 30 ft-lb)

1C442

b. Room-Temperature Aged 48 Days Following
   -T7 Heat Treatment

   (Impact energy = 5 ft-lb)

FIGURE 25. APPEARANCE OF FRACTURE SURFACES ON CAST CHARPY IMPACT
SPECIMENS AFTER -T7 HEAT TREATMENT

60

Conclusions

Results show that:

(1) Room-temperature age hardening will occur in material water quenched after heat treatment at temperatures as low as 250 F.

(2) Material air cooled from 350 F will also experience some hardening after room-temperature aging, but hardness appears to stabilize after 72 hours.

(3) Mechanical properties of material air cooled from 350 F will also experience changes after room-temperature aging but overall sheet formability is not impaired.

These studies have shown that the -T7 condition specified for LA141 alloy is, in fact, not a true stabilizing treatment since some precipitation hardening will occur upon aging at room temperature. However, this increase in hardness does not impair the properties of vacuum-induction melted material. Until such time that this quality material becomes commercially available, it is recommended that the -T7 stabilizing heat treatment consist of 24 to 48 hours at 200 F or a somewhat shorter time increment at 225 to 250 F followed by air cooling.

XI

SCRAP-RECOVERY STUDIES

One of the major objectives of this program was the establishment of techniques for recycling of drossy scrap, machine turnings, sheet trimmings, etc., since present commercial production methods for Mg-Li alloys utilize only a very small percentage of the scrap as remelt material during casting. The best-known approach to the recovery of scrap material is the use of refining fluxes which will wet the metal and the trapped impurities, and, by virtue of density differences, separate the impurities from the metal.

Two known flux compositions served as the basis for this study. In previous studies, [5] Battelle developed and used a 75 percent LiCl-25 percent LiF flux composition very successfully. In the casting of 1300 pound ingots by Dow, [4] a 50 percent KCL-37.5 percent LiCl-12.5 percent LiF flux was used.

Ingot sizes ranging from 3 to 80 pounds were cast. The use of screens or filtering techniques was included in the study as a further attempt to separate dross and other impurities from the melt.

Initial Examination of Flux Behavior During Melting

The LiCl-LiF and KCl-LiCl-LiF fluxes mentioned above (plus variations of these compositions) were investigated, along with flux compositions suggested by Dow Chemical Company and Foote Mineral Company as well as those developed by Battelle. Since the exact compositions of these fluxes are considered company confidential, they will be noted as Dow 1, Dow 2, Foote 1, or modifications thereof. The Battelle developed fluxes are designated as Dow/Battelle 1, Dow/Battelle 2, etc.

Casting Studies

Initially, three 6-pound heats were cast from scrap material that remained from previous Battelle studies.

Melting was done in a gas-fired pot furnace with melt weights of approximately 6 pounds.

The melting practice used was essentially the same as that used for melting virgin alloy and is described briefly here:

(1) Premelt enough flux in a steel crucible to give a total melt consisting of 25 percent flux, the rest alloy.

(2) Charge the required amount of solid scrap to the molten flux.

(3) Stir the mixture vigorously and allow a 15-minute settling period. During this period, a thin layer of flux on top of the molten alloy protects it from the atmosphere, while the remainder of the flux settles to the bottom of the crucible.

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(4) Remove the flux film, which becomes somewhat viscous during the
settling period, and pour by decanting the melt from above the
bottom flux layer.

The alloys were chill cast in rectangular steel molds by a modification of the
Durville process. That is, the mold was tilted about 45 degrees from the vertical to
receive the melt, which was poured slowly to avoid turbulence. As pouring progressed,
the mold was rotated to a vertical position. Immediately after casting, a steel plate
heated to redness was placed over the top of the mold to minimize pipe formation and
also to exclude air from the solidifying ingot. Water was then sprayed on the bottom of
the mold to promote directional solidification.

The scrap form and condition and flux composition were varied in these three
heats as shown below:

Heat	Type of Charge	Pickled Scrap	Flux Type
1	Plate and extruded sections	No	75LiCl-25LiF
2	4.35 lb plate scrap plus 1.6 lb foil scrap	No	50KCl-37.5LiCl-12.5LiF
3	Plate and extruded sections	Yes	75LiCl-25LiF

Observations of each heat during melting and casting indicated that:

(1) The LiCl-LiF flux remained generally fluid throughout

(2) The KCl-bearing flux became extremely viscous during meltdown
and argon was needed to prevent burning after the flux was skimmed

(3) Pickling of the scrap eliminated essentially all dross formation

(4) Compaction of chips and thin foil scrap will be necessary before
melting to prevent excessive burning.

Listed below are the metal yields from each ingot:

Heat	Charge Weight, pounds	Cast Weight, pounds	Cropped Ingot Weight, pounds	Metal Recovery, percent
1	6	5.50	5.00	83.3
2	6	4.20	3.95	66.0
3	6	5.30	4.73	79.8

It is interesting to note that pickling of the scrap did not appear to improve metal yield.
However, the ultimate ingot quality should be better where the cleaner scrap is used.
The low metal recovery in Heat 2 is due in part to the burning that occurred during
charging of foil scrap and chips. It is also likely that some metal was trapped in the
viscous slag of this heat.

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The three flux-melted ingots are shown in Figure 26.
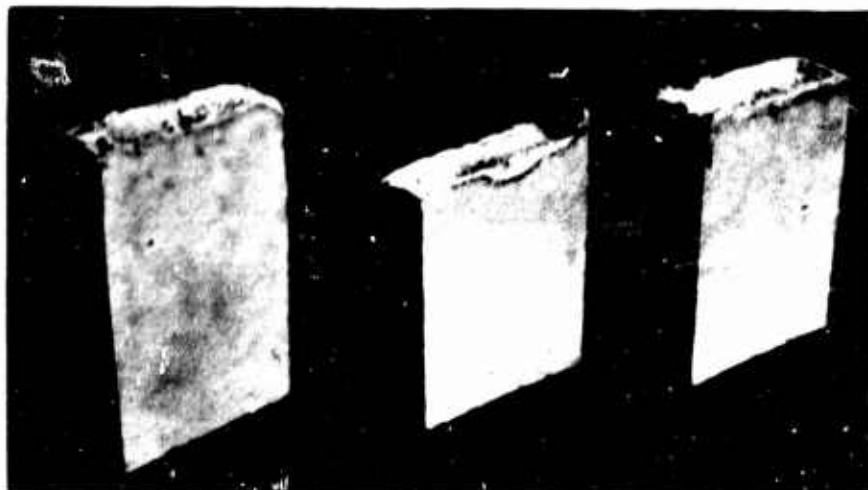
33811

FIGURE 26. THREE FLUX-MELTED INGOTS OF LA141 ALLOY

Following the apparent success with the small heats, 20-pound heats were cast using the LiCl-LiF flux. For this work, sheet and cast ingot tops were supplied by Brooks and Perkins. For Heat 4, only the loose oxide was brushed from the scrap material prior to melt down. As melting progressed, dross formation became excessive and considerable burning occurred. The addition of extra flux (total of 33 percent of the melt weight) and the use of an argon blanket failed to improve conditions.

The severity of dross formation is evidenced by the 5-1/4 pound recovery obtained from the original 20-pound heat. These results indicated that:

(1) Pickling of scrap was necessary, contrary to previous results with 6-pound heats.

(2) New equipment modifications were needed to handle melting and casting operations.

This ingot was remelted in subsequent scrap-recovery studies.

Prior to casting of an additional 20-pound heat, a sheet-metal hood with asbestos-cloth drop curtains was constructed over the melting furnace. A cylindrical crucible "extension" was fabricated that could be in place on top of the crucible during melt down to provide a deep argon mantle and still be removable for pouring. A more efficient ventilating system was installed for fume removal. A positive pressure of argon was maintained by forcing argon through a rectangular box which was perforated on the side facing the melt. This box covered the top of the crucible.

Figure 27 shows the melting furnace with crucible and argon box in place. Figure 28 shows the melting area including furnace, hood, and casting unit.

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FIGURE 27.   MELTING FURNACE USED FOR
SCRAP-RECOVERY STUDIES
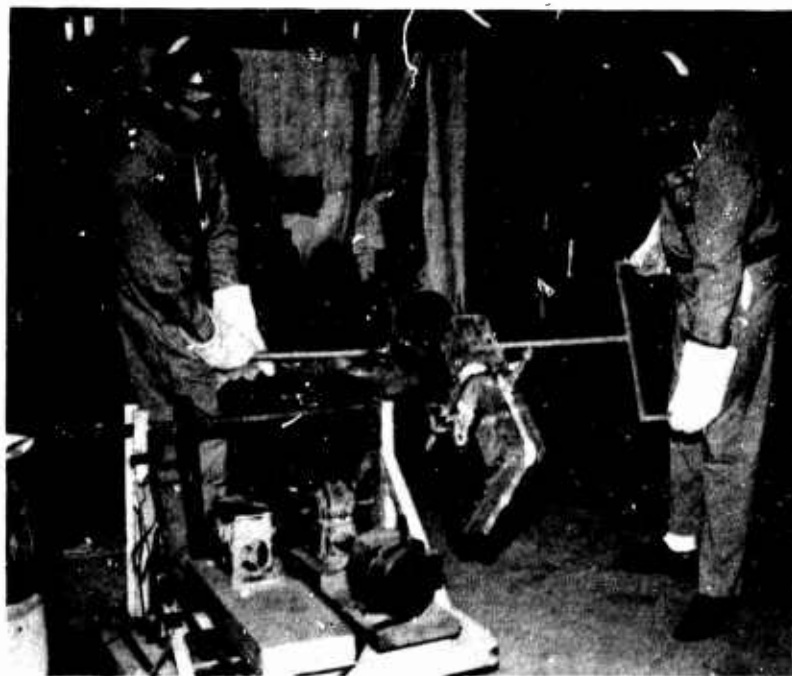
36924

FIGURE 28.   MELTING AND CASTING AREA USED FOR
SCRAP-RECOVERY STUDIES

Scrap material was pickled in the following solution:

> 24 oz chromic acid
> 5-1/2 oz ferric nitrate
> 0.47 oz potassium fluoride
> Sufficient water to make 1 gallon of solution.

This solution easily removed the dross and oxide and left the metal with a bright surface finish.

For Heat 5, pickled scrap was melted and 2 pounds of LiCl-LiF flux (10 percent of initial charge weight) was added to clean the melt. Dross formation was not excessive and very little burning occurred. An 18.1-pound ingot was subsequently cast without difficulty. Evaluation of this material is described in a later section of this report.

Evaluation of New Fluxes

Nine new flux compositions were evaluated in the casting of 6 to 20 pound heats of LA141 scrap using procedures outlined previously. Three of these flux compositions were suggested by Dow Chemical Company. Foote Mineral Company supplied a fourth flux material for evaluation. The remaining compositions were developed by Battelle based in part on initial Dow flux compositions.

The exact compositions of these fluxes are considered proprietary information by the respective companies but are supplied to the Air Force in a supplemental attachment to this report.

The results obtained with these fluxes are summarized below:

Heat	Flux Designation	Flux Added, percent of total charge	Flux/Metal Separation	Burning During Melting	Metal Recovery, percent of original charge
18	Dow 1	27	Very Good	Slight[b]	76
19	Dow 2	20	Good	Very Slight	91
21	Dow/BMI 1	20	Very Good	Normal	84
23	Foote 1	20	Fairly Good	Considerable	64[a]
24	Dow 3	25	Fairly Good	Normal	69
25	Dow/BMI 2	25	Good	Normal	79
26	Dow/BMI 3	25	Good	Slight[b]	80
27	Dow/BMI 4	25	Very Good	Slight[b]	85
28	Dow/BMI 5	27	Good	Normal	74

(a) Metal spilled during the pour.
(b) More than normally encountered.

The metal recoveries reported in Heats 24-28 in some instances are lower than were obtained with flux compositions tried previously. However, these results reflect the use of dirty scrap as part of the starting charge material. This reduces the degree of flux-metal separation that is obtained during melt down and thus lowers metal recovery from the melt.

The best overall flux "performance" was obtained with Dow/Battelle Fluxes 4 and 5. With Flux 5 (Heat 28), a 13.5-pound charge of uncleaned, compacted machine turnings and chips was melted using a flux/metal ratio of ~27 percent. Metal recovery was 74 percent, which is very good considering the condition and form of the starting material. This flux was subsequently used in the casting of a 90-pound scrap heat which is described below.

Casting of a 90-Pound Scrap Heat

The casting studies involving various flux compositions have been described above. The results have shown several fluxes to be capable of removing contaminants from scrap heats and providing good potential recovery of Mg-Li metal.

Based on these results, it was decided to melt and cast a 90-pound flux heat using the same equipment that was available for the vacuum-induction casting program. The Dow/Battelle Flux 5 was selected for this work based on the results of the smaller heats.

The melting and casting cycle for this heat is described below:

(1) Charge 90 pounds of clean (pickled) scrap and 15 pounds of Dow/Battelle Flux 5.

(2) Cover crucible with perforated box to provide positive pressure of argon.

(3) Begin melting cycle under argon flow of 2 cu ft/hour.

(4) When charge is melted, stir for 5 minutes.

(5) Allow settling period of 15 minutes.

(6) Heat to 1300 F.

(7) Pour ingot in air into mold purged with argon.

Considerable fuming occurred at the outset of melting but this diminished as the flux became molten. Some delay in the melting cycle was encountered due to bridging of some of the charge material in the top of the crucible. In any event, melt-down time was much longer than encountered in vacuum-induction melting of a comparable size charge.

The hinged stirring plate used to agitate the vacuum-induction melts was also used here for stirring. During retraction of the stirring mechanism from the melt, the plate became detached from the rod and dropped back to the bottom of the melt. Stirring had been completed, however, when this occurred.

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After the settling period, the ingot was poured without difficulty. Solidification shrinkage was confined to the top 2 to 3 inches of the ingot. The cast ingot weighed 80.5 pounds, for 89.5 percent recovery.

Chemical analyses made on the top and bottom of the cast ingot are reported as follows:

Ingot Location	Composition, percent	
	Lithium	Aluminum
Top	13.8	1.38
Bottom	13.8	1.32

Assuming that the starting charge did not contain any "off-analysis" material, the lithium and aluminum recoveries were excellent. Indications are that scrap material could be remelted without virgin melt additions to obtain the proper alloy compositions.

This ingot was sectioned for examination and considerable flux entrapment was noted. One very large flux area near the base of the ingot is known to be due to some flux becoming lodged under the pouring spout and being poured out into the mold at the start of pouring.

In general, the ingot quality looked quite good for the first attempt at casting a large ingot, and the problems of flux entrapment appeared to be primarily due to equipment or operational deficiencies rather than problems with metal-flux separation. Since extensive changes would be required to modify existing equipment to cast additional heats, no further large ingots were cast.

Optimization of Stirring and Settling Periods
for Flux Heats

In preparation for the flux melting and casting of the 90-pound scrap heat of LA141 alloy described previously, a brief study was made to examine the required stirring and settling periods that were necessary to clean the melt and provide for separation of the flux from the melt prior to pouring. A 20-pound scrap heat was melted for this work. Determination of the minimum flux-to-metal ratio required to clean the metal using the Dow/Battelle Flux 5 was included in the study.

The scrap charge was melted originally without any flux. Flux additions were made, and the melts were stirred for 1 to 5 minutes. Samples were extracted from the melt and poured into graphite molds after settling periods that varied to 20 minutes.

Ten samples were obtained during this study. The sample conditions are defined in Table XV. The last sample was taken from the 13-pound ingot cast at the conclusion of the work.

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TABLE XV. SAMPLE SEQUENCES FOR EVALUATING REQUIRED
STIRRING AND SETTLING PERIODS IN FLUX-
MELTED SCRAP HEATS OF LA141 ALLOY

Sample	Ratio of Flux Weight/ Total Charge Weight, percent	Total Stirring Time, minutes	Settling Period, minutes
0	0	0	0
1	0	0	0
2	5	1	1
3	5	1	5
4	13	2	1
5	13	2	5
6	13	2	15
7	13	5	1
8	13	5	5
9	13	5	~20

A microstructural examination of the as-cast samples was used to evaluate the effects of flux quantity, stirring time, and settling period on the cleanliness of each sample. The results showed that, once sufficient flux was present, a stirring time of 3-5 minutes combined with a settling period of at least 10 minutes was the optimum treatment.

It is interesting that these cycle times correspond closely with those used years ago in early flux-melting studies with these alloys. Apparently, the contaminants present in the melt when cleaned scrap is used are not of sufficient quantity to severely alter the procedures used formerly with virgin starting material.

Evaluation of Screening Materials

In addition to the use of fluxes for removing impurities from remelted scrap, investigations were made of various screening or filter materials for retaining impurities during ingot pouring. To accomplish this, the filtering media must trap small solid particles but pass the molten metal through at a sufficient pouring rate to prevent freezing.

For this portion of the study, commercially generated LA141 alloy scrap was supplied by Brooks and Perkins. All scrap was pickled in a ferric nitrate solution prior to melting under an argon atmosphere. Tests were made both with and without the addition of flux. Two types of filtering techniques were employed, each using various materials as indicated below:

Screening devices:
    (1) Glass-fiber screen (12 mesh)
    (2) Glass wool
    (3) Steel wool
        (a) Coarse
        (b) Fine
    (4) Perforated metal screen (40 holes/square inch)
        (a) Inconel
        (b) Nickel

Filter bed:
   (1) Steel shot (1/16-inch diameter)
   (2) Glass beads (6-millimeter diameter)
   (3) Steel balls (3/16 to 1/4-inch diameter)

Four of the screening materials tried are shown in Figure 29. All filter materials were positioned in a graphite funnel that was mounted above a rectangular steel mold. A photograph of the graphite funnel is shown in Figure 30. Screens were fit into the slot shown in the photograph. Bed-type materials were packed in the funnel opening.

The LA141 alloy scrap was melted in a steel crucible, heated to 1350 to 1500 F under an argon atmosphere, and poured in air.

Initially, none of the filtering material — screen or bed type — would allow the molten metal to flow. The molten alloy formed a tight film on contact with the filter material and plugged the filter. Heating the molten metal to high temperatures and preheating the filter had little effect. An air-driven vibrator attached to the filter stand, when operated at high enough frequency, caused the molten metal to flow through the screen-type filters. No metal penetration of the bed-type materials was obtained, however. Typical results with these materials are shown also in Figure 30.

Metallographic examination of the cast ingots revealed that inclusions were more prevalent in this material than with ingots poured without filtering. The filtering media created more surface area in the metal during pouring and subsequently more surface film or oxide that broke up and went directly into the melt. As a result of the poor quality obtained with this cast material, no further efforts were made in this area. As indicated previously, the use of fluxes with appropriate skimming prior to and during pouring represents the best approach to scrap recovery to date.

Ingot Fabrication

All ingots cast in the study of scrap-recovery techniques were fabricated by similar methods. The as-cast ingots were grit blasted, homogenized for 20 hours at 500 F, and broken down to slabs approximately 1-3/4 inch thick. After reheating they were cross rolled to increase the original ingot width by 50 percent and then reduced to 0.063-inch-thick sheet. With the exception of the cross rolling mentioned, the material was rolled parallel to the long axis of the original ingot.

Like to many other materials, Mg-Li alloys may check during breakdown rolling. To avoid such defects, light passes of 5 to 10 percent reduction per pass were taken until the slabs were reduced to 1-3/4 inch. Slabs were then rolled to 0.063-inch-thick sheet with reductions in thickness of 20 percent per pass. The material was returned to the furnace and reheated to 500 F between each pass.

In some instances where the cast quality was not too good and numerous surface defects appeared during rolling, the slabs were machined on top and bottom surfaces while still in plate form (1/4 inch to 1 inch in thickness).

The fabrication behavior of these ingots was observed during rolling as a portion of the overall evaluation of cast-ingot quality. Further evaluations of mechanical properties, formability, and material cleanliness were made on rolled sheet and are described below.

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STAINLESS STEEL SCREEN
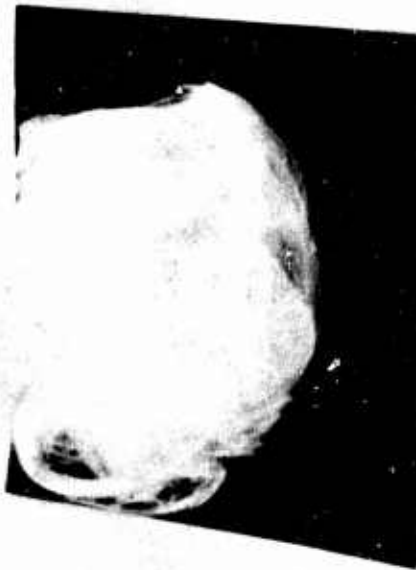
STEEL WOOL · ·

GLASS SCREEN ·

GLASS WOOL ·

FIGURE 29. SCREENING MATERIALS EVALUATED FOR REMOVAL OF IMPURITIES DURING
CASTING OF LA141 ALLOY SCRAP HEATS

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Screen
positioned in
horizontal
slot

GRAPHITE CONE FILLED
WITH 6 MM GLASS BEADS

35619

FIGURE 30. GRAPHITE FUNNEL USED FOR EVALUATION OF SCREENING
AND FILTER-BED TYPE MATERIALS FOR REMOVING
IMPURITIES DURING POURING

Photograph also shows results of attempts to pour LA141
through glass bead filter media.

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Evaluation of Sheet Quality

Excluding the material cast in evaluating screening materials, a synopsis of material cast, fabricated, and evaluated in the scrap-recovery studies is listed below:

Heat	Melt Size, pounds	Cast-Ingot Weight, pounds	Melting Flux	Quality Evaluations Conducted			
				Rolled	Mechanical Properties	Bend Tests	Microstructure Examination
1	6	5.5	LiCl-LiF	Yes	Yes	Yes	Yes
2	6	4.2	KCl-LiCl-LiF	Yes	Yes	Yes	Yes
3	6	5.3	LiCl-LiF	Yes	Yes	Yes	Yes
5	20	18.1	LiCl-LiF	Yes	Yes	Yes	Yes
18	4	3.1	Dow 1	Yes	Yes	Yes	Yes
19	6	5.5	Dow 2	Yes	Yes	Yes	Yes
21	6	5.0	Dow/BMI 1	Yes	Yes	Yes	Yes
23	6	3.9	Foote 1	Yes	Yes	Yes	Yes
24	6	4.2	Dow 3	Yes	Yes	Yes	Yes
25	6	4.8	Dow/BMI 2	Yes	Yes	Yes	Yes
26	6	4.8	Dow/BMI 3	Yes	Yes	Yes	Yes
27(a)	6	5.1	Dow/BMI 4	No	No	No	Yes
28(a)	13.5	10.0	Dow/BMI 5	No	No	No	Yes
29(a)	20	15.0	Dow/BMI 5	No	No	No	Yes
30	90	80.5	Dow/BMI 5	No	No	No	Yes

(a) No fabrication studies planned as flux compositions vary only slightly from Heat 25.

Mechanical-Property and Bend-Test Data

The quality of sheet material fabricated from the first series of flux-melted scrap heats of LA141 alloy was evaluated by bend-test and mechanical-property determinations. Results are summarized in Table XVI and show that properties of Heats 1 and 3 were very good. Strength levels were good for all materials, but Heat 2 showed lower elongation values. Sixteen 1T bend tests were made on material from each heat. The 1T bend-test results correlate with mechanical properties and are indicative of good-quality material.

TABLE XVI. MECHANICAL-PROPERTY AND BEND-TEST DATA ON AS-ROLLED 0.063-INCH-THICK SHEET FABRICATED FROM 6-POUND FLUX-MELTED INGOTS OF LA141 ALLOY (HEATS 1, 2, AND 3)

| | | | | | Mechanical Properties | | | |
| | | | | | Ultimate Tensile Strength, 1000 psi | Yield Tensile Strength, 1000 psi | Elongation, percent in 2 inches | |
Heat	Melting Flux	Scrap Charge Cleaned	Number of Tests	Specimen Orientation				Summary of Transverse 1T Bend Tests
1	LiCl-LiF	No	4	L	19.4-20.3	16.8-17.2	51-64	OK-show "orange peel"
			4	T	22.4-22.5	18.6-19.6	28-40	
2	KCl-LiCl-LiF	No	4	L	24.9-25.2	23.4-23.8	27-29	Fractured
			4	T	27.6-27.9	25.8-26.2	10-13	
3	LiCl-LiF	Yes	4	L	26.1-26.6	19.3-20.4	49-70	OK
			4	T	25.0-25.2	20.0-20.6	35-46	

Similar data on Heat 5, the first 20-pound ingot cast, are reported in Table XVII. Results again indicate that good-quality material was obtained.

TABLE XVII. MECHANICAL-PROPERTY DATA ON AS-ROLLED 0.063-INCH-THICK SHEET FABRICATED FROM A 20-POUND FLUX-MELTED INGOT OF LA141 ALLOY (HEAT 5)

| | | Mechanical Properties | | | |
Number of Tests	Specimen Orientation	Ultimate Tensile Strength, 1000 psi	Yield Tensile Strength, 1000 psi	Elongation, percent in 2 inches	Summary of Transverse 1T Bend Tests
10	L	19.3-20.1	16.4-17.8	21-35	OK
10	T	20.1-21.6	17.7-18.9	6-20	OK

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The quality of sheet material fabricated from Heats 18, 19, 21, and 23 to 26 was also evaluated by bend tests and mechanical-property determinations. Results summarized in Table XVIII indicate that the presence of KCl in the flux appears to have an adverse effect on sheet ductility. Specimens from material originally cast with KCl-bearing fluxes fractured during the standard 1T bend tests. Other sheet properties were typical of those normally obtained with the LA141 alloy.

Material from Heats 27 and 28 were not fabricated since flux compositions varied only slightly from Heat 25 and observations during casting indicated that good-quality material was produced. The composition changes were made to reduce the potential cost of the flux materials being used.

TABLE XVIII. MECHANICAL-PROPERTY AND BEND-TEST DATA ON 0.063-INCH-THICK SHEET[a] FABRICATED FROM FLUX-MELTED INGOTS OF LA 141 ALLOY

| Heat | Melting Flux | Number of Tests | Specimen Orientation | Mechanical Properties | | | Results of 90-Degree Bend Tests over 1T Die |
				Ultimate Tensile Strength, 1000 psi	Yield Tensile Strength, 1000 psi	Elongation, percent in 2 inches	
18	Dow 1[b]	4	L	19.6-19.9	15.9-16.3	27-31	Fractured
19	Dow 2[b]	6	L	19.5-19.9	15.7-16.1	26-31	Fractured
21	Dow/BMI 1	6	L	20.1-20.2	16.2-16.5	30-34	OK
23	Foote 1	6	L	19.6-19.9	15.7-15.8	25-33	OK
24	Dow 3[b]	3	L	19.4-19.6	15.1-15.3	35-39	OK
		3	T	21.1-21.2	17.0-17.3	21-22	Fractured
25	Dow/BMI 2	3	L	18.8-19.0	14.2-14.4	36-42	OK
		3	T	19.8-19.9	14.9-15.0	32-45	OK
26	Dow/BMI 3	3	L	21.1-22.0	18.1-19.3	27-35	OK
		3	T	22.9-23.4	19.7-20.2	26-31	Fractured/OK

(a) Commercial -T7 heat treatment of 6 hours at 350 F, air cooled.
(b) KCl-bearing flux.

Metallographic Examination

Microstructure examination was used throughout these studies to attempt to evaluate cleanliness of the cast ingots. Several representative examples are shown here to illustrate some of the variations that have been observed. Figure 31 shows the effect of "dirty-versus-cleaned" scrap in Heats 2 and 3, respectively.

Figure 32 shows some microstructures from cast samples of Heats 16, 19, 21, and 23. These photographs show variations in particle content from heat to heat. Some of these particles are known to be the Al-Li phase - particularly those in evidence in the grain boundaries. True differences among materials from these heats were not revealed in subsequent property and formability tests. The apparent effect of KCl in the flux on formability was not detected in any microstructure examinations.

75

100X   9A898

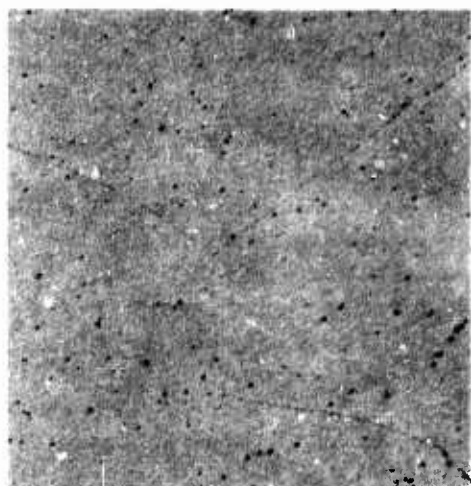Heat 2 - Dirty Scrap

100X   9A899

Heat 3 - Cleaned Scrap

FIGURE 31. UNETCHED MICROSTRUCTURES OF LA141 ALLOY SHOWING
EFFECTS OF CLEANED SCRAP ON CLEANLINESS OF ROLLED
SHEET
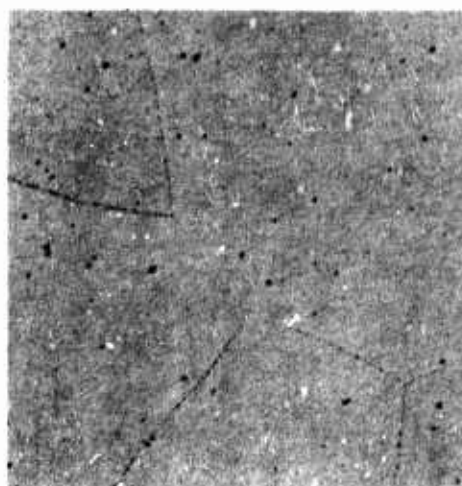
100X   4B618   100X   4B617
Heat 18-Dow Flux   Heat 19-Dow Flux
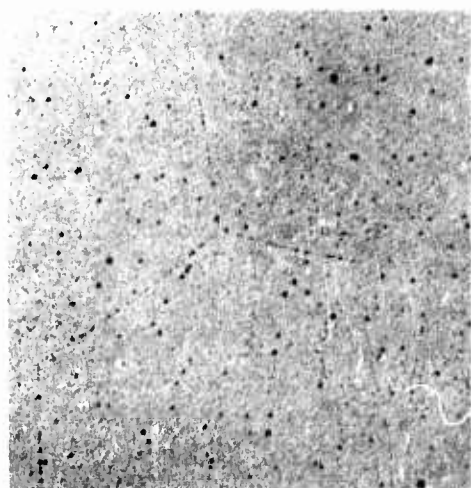
100X   4B620   100X   4B619
Heat 21-Dow/Battelle Flux   Heat 23-Foote Flux

FIGURE 32. AS-CAST MICROSTRUCTURES OF LA141 ALLOY CAST WITH
VARIOUS MELTING-FLUX COMPOSITIONS

Samples etched in 75 Glycol-24H_2O-1HNO_3 solution.

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Technique for Evaluating Cast Quality of "Scrap" Ingots

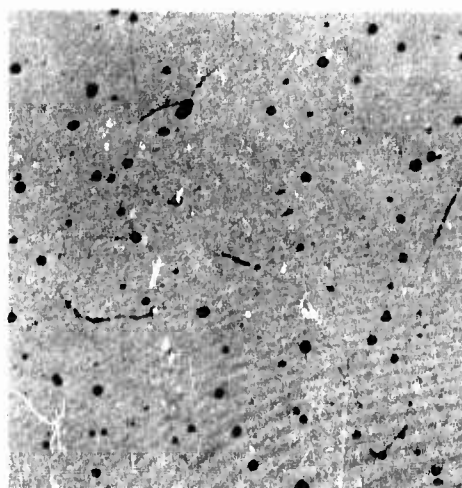A brief study conducted after the conclusion of the evaluation program on wrought material from the scrap-recovery studies suggests that Charpy impact tests could provide a good clue to variations in ingot quality in future studies. Table XIX shows the results of Charpy impact tests on cast material from several flux-melted heats along with data on Heat G — the best vacuum-induction melted material produced in this program.

Although the data are not conclusive (based on only limited tests), the results do show that the high impact values obtained on cast material tend to correlate with indications of good-quality material obtained in mechanical-property determinations, bend tests, etc. This technique could find particular use as a screening method in preliminary evaluations of cast-ingot quality.

TABLE XIX. RESULTS OF CHARPY IMPACT TESTS ON
AS-CAST LA141 ALLOY MATERIAL

Heat	Melting Flux	Impact Energy[a] ft-lb	Bend Ductility of Wrought Material over 1T Die
18	Dow No. 1	12.5/13.5	Fractured
21	Dow/BMI No. 1	34.0/39.5	OK
23	Foote No. 1	17.0/17.0	OK
24	Dow No. 3	21.0/24.0	Fractured
25	Dow/BMI No. 2	29.5/36.0	OK
26	Dow/BMI No. 3	32.0/30.5	OK/fractured
G	Vacuum-Induction Cast (No Flux)	35.0/32.0	OK

(a) Cast material given the -T7 heat treatment and tested immediately after heat treatment.

Estimated Flux Costs for Recovery of Mg-Li Scrap

In the course of the study of flux compositions that would be effective in the recovery of scrap, attention was given to the ultimate costs of using these fluxes. In fact, the last fluxes evaluated were modifications of previous compositions in which less costly additives were used to replace certain lithium compounds which were known to be expensive.

Estimates of the cost per pound of all the fluxes evaluated in this program were ultimately calculated based on car-load prices of the individual compounds used in the flux makeup. These costs are listed in Table XX.

TABLE XX. COST ESTIMATES OF FLUXES USED IN
SCRAP-RECOVERY STUDIES

Flux Designation	Approximate Flux Cost, dollars per pound
LiCl-LiF	1.01
KCl-LiCl-LiF	0.51
Dow 1	0.44
Dow 2	0.55
Dow 3	0.43
Foote 1	7.10
Dow/BMI 1	0.84
Dow/BMI 2	0.84
Dow/BMI 3	0.96
Dow/BMI 4	0.73
Dow/BMI 5	0.81

The low costs of the Dow fluxes reflect the extensive use of KCl which is quite inexpensive. The use of KCl was indicated to be detrimental to the properties obtained on material cast with KCl-bearing fluxes, although this effect may not be carried over in material subsequently remelted and diluted in the production of commercial ingot.

The costs of Dow/Battelle Fluxes 4 and 5 reflect the changes made in Flux 3 to reduce the overall cost, which is ~20 to 25 percent less than that of the LiCl-LiF compositions used in earlier work. Assuming that the ratio of "flux-to-metal" charged in the scrap recovery process is 1:3, then the actual flux costs will be about 25-26 cents per pound of scrap material remelted.

Conclusions

Based on the results of the scrap-recovery studies it is apparent that several flux compositions show promise in successfully reclaiming Mg-Li alloy scrap to provide a good remelt ingot that can be used as charge material in the vacuum-induction process.

These fluxes have been developed with the intent of providing a flux material that will do the intended job as inexpensively as possible. In the Dow/Battelle fluxes, the expensive ingredients have been reduced in quantity or eliminated entirely with good success. While the results with the 90-pound ingot were not a total success, the studies conducted across the entire range of ingot sizes have shown that scrap recovery is feasible and with further development should become an important factor in reducing the end cost of Mg-Li mill products.

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XII

COMPARISON OF CORROSION RATES FOR FLUX-MELTED AND VACUUM-INDUCTION MELTED MATERIAL

A limited number of corrosion tests were made to compare sheet samples from flux-melted scrap Heats 1, 2, 3, and 5 and vacuum-induction-melted Heats C and D. From 6 to 8 sheet specimens from each heat, measuring $3 \times 1\text{-}1/2 \times 0.063$ inch, were submitted for exposure to a 100 percent relative humidity atmosphere at 95 F for 30 days. Specimens were lightly abraded with 600-grit paper, cleaned, weighed, and sheet-thickness recorded prior to testing.

After exposure all specimens were mottled with a light gray oxide. The humidity cabinet was opened to allow the specimens to drip dry for 2 to 3 hours. They were then placed in a desiccator containing silica gel for 80 hours. The specimens were weighed and placed in an oven at 180 F for 1 hour prior to reweighing to determine the moisture content of the oxide.

The weight gains after oven drying were evaluated by analysis of variance with the following results:

Heat	$\overline{X}$, g	$2S_m$
1	+0.3385	±0.0397
2	+0.3299	+0.0397
3	+0.3266	±0.0397
5	+0.3619	±0.0458
C	+0.3884	±0.0458
D	+0.4334	±0.0424

The standard deviation on the true means (S_m) were calculated from the best estimate of the variance (S^2) on all the replicate specimens and the number of specimens in each heat.

The corrosion products were chemically removed in a 20 percent by weight chromium trioxide - 1 percent silver nitrate water solution. The specimens were exposed in the boiling solution for approximately 30 seconds and then immediately rinsed in cold water. After a second rinse in boiling distilled water, the specimens were dried in an oven for 2 hours at approximately 180 F, and then weighed. The weight losses and analysis of variance are reported below:

Heat	$\overline{X}$, g	$2S_m$
1	-0.1613	±0.0294
2	-0.1562	±0.0294
3	-0.1743	±0.0294
5	-0.1534	±0.0348
C	-0.1650	±0.0348
D	-0.2226	±0.0318

The standard deviations on the true means (S_m) were again calculated from the best estimate of the variance (S^2) on all the replicate specimens and the number of specimens in

each heat. These data indicate no significant difference among Heats 1, 2, 3, 5, and C or among heats 3, C, and D. However, D appears to be significantly different from the other heats.

Heats	$\overline{X}$, g	$2S_m$
1,2,3,5,C,	-0.1624	±0.0131
D	-0.2226	±0.0318

A review of the analysis of variance for the weight gains indicates a similar behavior.

Heats	$\overline{X}$, g	$2S_m$
1,2,3,5,C,	+0.3462	±0.0064
D	+0.4334	±0.0424

Assuming the corrosion product is adherent and is principally $Mg(OH)_2 \cdot H_2O$, these weight gains are equivalent to the following weight losses:

Heats	X, g	$2S_m$
1,2,3,5,C,	-0.162	±0.003
D	-0.201	±0.020

If it is further assumed that the corrosion rates are constant with time, these weight losses can be converted to the following metal corrosion rates in $mg/dm^2/day$:

Heat numbers		Corrosion rate, mdd
1,2,3,5,C	from weight gain	8.7
	from weight loss	8.75
D	from weight gain	10.8
	from weight loss	12.1

Figure 33 shows the specimen appearance after testing and after the corrosion product has been removed. The pits on the specimens are very shallow (maximum 3 mils) and cover approximately one fourth of the surface. An average pit depth of 2 mils could account for more than half of the weight loss. It is believed that the pit growth was stifled by the buildup of corrosion products and that a much longer period of exposure would show a more general roughening of the surface.

The corrosion rates reported above are similar to those reported previously by Battelle[5] for the LA141 alloy. Of particular interest are the good results obtained on the flux-melted scrap heats which compare favorably with material cast by the vacuum-induction technique. The cause of the difference between heat D and the other five heats is not known.

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3X   C2608   3X   C2609

    a. As-Exposed Surface   b. Surface After Chemical
                                                 Cleaning

FIGURE 33. APPEARANCE OF SURFACE OF LA141 ALLOY
                SHEET AFTER EXPOSURE FOR 30 DAYS AT 95 F AND
                100 PERCENT RELATIVE HUMIDITY

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APPENDIX I

STATE-OF-THE-ART SURVEY OF MELTING, CASTING, AND FABRICATING MAGNESIUM-LITHIUM ALLOYS

Magnesium-lithium alloys react readily with oxygen, nitrogen, and moisture in the temperature range used for fabricating the alloys. Thus, it is essential to protect the molten metal during melting and casting. An inert-gas atmosphere maintained both during melting and casting has shown the greatest promise for production of sound, clean ingots. Protection of ingots during heat treatment and metal working operations is less important. In the solid state, the alloys tend to form an oxide coating that protects the metal from further reaction with atmospheric gases.

MELTING AND CASTING

Magnesium-lithium alloys have been melted by various techniques. Melt sizes have ranged from a few pounds to 13,000 pounds. However, all the techniques have incorporated some type of protection. Chief among the protective media used have been fluxes and inert gases.

As with melting, molten magnesium-lithium alloys also require protection during casting. For this purpose, inert gases have been exclusively used. Ingot-casting techniques have included the Durville process, a mold-withdrawal process, a bottom-pouring process, and a pressure-pouring process.

The melting and casting of magnesium-lithium alloys has been extensively covered in the literature. The following survey describes the six major techniques indicative of industrial potential.

Battelle Flux Method

The earliest description[9] of a satisfactory method for melting and casting magnesium-lithium alloys was reported by Battelle in 1949. In this work, heats weighing 10 pounds or more were melted under a flux or an inert atmosphere. The flux consisted of lithium chloride and lithium fluoride generally in the ratio of 75 to 25. This ratio was recommended for melting new types of alloys for the first time. However, a ratio of 82 to 18 was used on certain alloys, especially those containing high percentages of cadmium.

In most of the experimental work, the quantity of flux used was equivalent to about 35 percent of the weight of the metallic charge. When "large" heats were made, that is, heats weighing 10 pounds or more, it was possible to reduce the percentage of flux.

A crucible having a diameter about one-third its height was used in preparing the melts. The flux was first melted to eliminate all traces of water from the hygroscopic flux ingredients. The magnesium and alloying elements other than lithium were

then added. In order to ensure that the alloying elements dissolved, they were placed in a steel wire basket suspended in the molten magnesium. Before the lithium was added, the molten flux was lighter than the metal and provided an effective protective layer on the melt. However, the introduction of lithium, added by means of an inverted cup plunged below the surface of the metal bath, reduced the density of the alloy to a value lower than that of the flux. Thus, the metal floated on top of the flux. Although the flux was heavier than the metal after the lithium addition, a thin film of flux remained on the surface. The protection offered by this film, combined with the stack effect of the deep crucible, reduced the tendency toward burning or excessive oxidation of the metal. Figure 34 depicts these melting conditions. In this figure, the crucible sketched has a well over which a steel cover may be held to hold back the flux during pouring. This was satisfactory for 10-pound heats but proved to be unnecessary when larger heats weighing over 100 pounds were made.

After all alloying additions were made, the flux was allowed to settle in the crucible. To increase the flux density and thus decrease settling time, additions to the flux were sometimes made. An addition of about 10 percent lithium bromide was found particularly effective.

The magnesium-lithium alloys were protected during pouring by a thin film of oxide which enveloped the stream. The Durville method of pouring, or a slight modification of it, was found necessary to preclude turbulence and attendant inclusion of the oxide skin in the ingot. Therefore, the molds were tilted in such a way that the metal ran down the side with a minimum of turbulence. (Figure 35). Ingot molds of either graphite or cast iron were used. When a cast-iron ingot mold was used, a hot top either of graphite or of a refractory baked at high temperature was needed. The surfaces of cast-iron molds were coated with acetylene soot. Before pouring, both graphite and cast-iron molds were heated to about 250 F to drive off moisture. After pouring, any tendency toward burning on the surface of the ingot hot top was eliminated by the use of G-1 powder (a commercial graphite-base material) or paraffin, or by covering with an air-tight lid. By following the procedure described, the magnesium-lithium alloys were found less likely to burn during melting and pouring than ordinary magnesium-base alloys.

Magnesium-lithium alloys were also melted under argon in a capped crucible without the employment of flux. In this method, the entire charge, including the lithium, was placed in a cold crucible. After purging the crucible with argon, the heat was melted and poured. Only a slight tendency toward burning during the pour was evident even though no salt flux was used.

A melting procedure combining the use of a flux and an argon atmosphere was also found to be highly satisfactory. Using this technique, the amount of flux required could be substantially reduced. The use of an argon atmosphere reduced oxidation and lessened the tendency for the flux to thicken. This combination of flux and argon atmosphere was subsequently used by Battelle to produce magnesium-lithium alloys for armo- [10] and aerospace[11] applications.

Although the Battelle flux method is satisfactory for melting and casting small magnesium-lithium ingots, the process has several disadvantages. For large heats the flux does not offer enough protection from burning and drossing. Also, since the flux and metal have only slightly different densities, it is difficult to obtain good separation
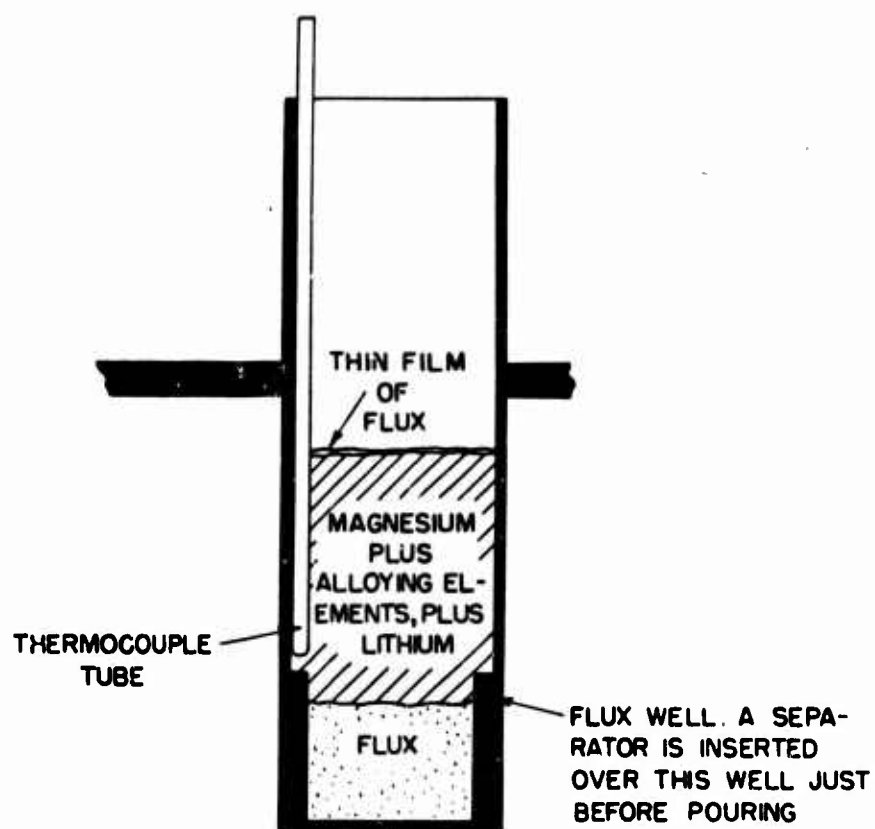
FIGURE 34. MELTING CRUCIBLE SHOWING RELATIVE POSITIONS
OF FLUX AND METAL AFTER LITHIUM ADDITION

85
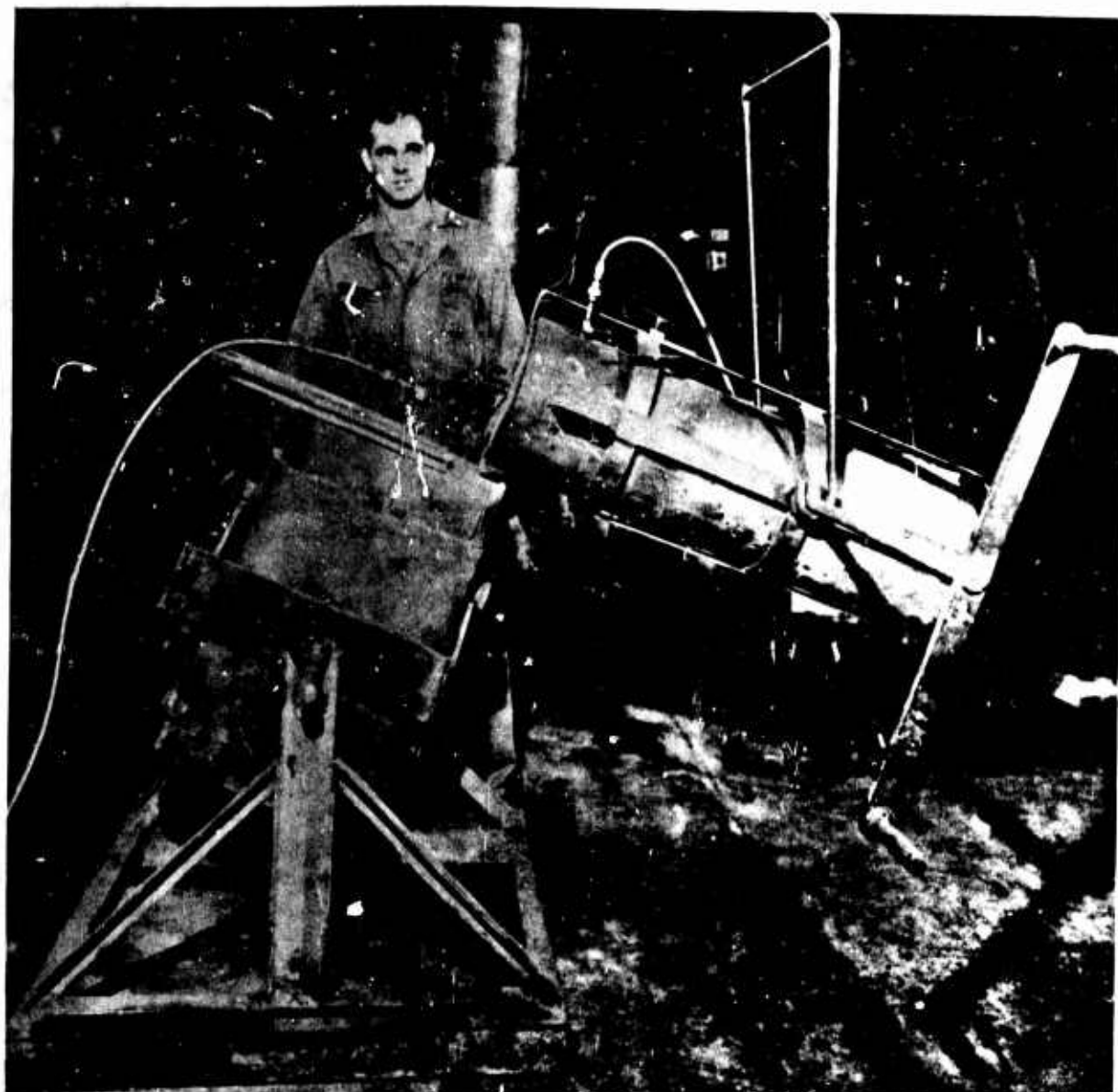
FIGURE 35. DURVILLE PROCESS FOR CASTING 100-POUND HEATS
OF MAGNESIUM-LITHIUM ALLOYS

Both crucible and mold are capped and have tubes for
introduction of argon gas atmosphere.

during pouring. Thus, flux inclusions in the ingot are a problem. The argon-gas-shield method, with or without flux, does not offer complete protection against oxidation, especially during the pour.

Dow Flux Method

In 1957, Dow Chemical Company developed[12] a production flux technique for melting and casting magnesium-lithium alloys. This work was performed for the U. S. Army Tank-Automotive Center, Detroit Arsenal. The alloys produced were used in fabricating an experimental armored hull for the M113 Armored Personnel Carrier (Figure 36).

Production melting and casting equipment used in the program was available at the Dow Metal Products Department's Madison, Illinois plant. The equipment included:

(1) Three 5,000-pound-capacity gas-fired furnaces and pots for melting and settling of molten metal

(2) One intermittent casting machine, including gas-fired holding pot, pump, molten-metal feed line and heaters, and pit area for use of either permanent mold or direct water-chill casting processes.

Heat sizes, ranging up to 13,000 pounds of alloy and 1300 pounds of flux, were the largest magnesium-lithium heats ever produced.

A specially prepared flux, NA No. 8166, was used. This had a composition by weight of 12.5 percent LiF, 37.5 percent LiCl, and 50.0 percent KCl. Lithium was added in the form of 5 and 10-lb ingots. Melt loss for all metals used was 8.1 percent. Continued vigorous stirring of the casting pot was required to obtain a relatively homogenous material.

The mold was designed to cast 11 by 27 by 86-inch slab ingots (1300 pounds). The bottom and 11-inch-wide sides were made of 3-inch steel plate. For the 27-inch-wide sides, 2-inch-thick steel plates were used. A 1/8-inch taper was used on all sides. The mold was equipped with a hot top in order to feed the slab. A 30-lb vibrator was mounted on each edge of the mold near the top. A mold withdrawal technique was used in order to lessen turbulence. Figure 37 shows an as-cast ingot.

As a safety precaution, the mold was mounted in a runout pan of equal capacity. The mold was bolted to the hydraulic elevator platform. Steel guide wheels were mounted on the floor plates to make the mold more stable by riding the mold faces during descent. A platform was necessary to enable the metal caster to see into the mold. The top of the mold, at the start of the descent, was 7 feet above the floor level.

An air-driven casting pump, using a protective atmosphere of helium, was used to transfer the metal to the mold through a 3/4-inch-diameter casting line. The casting line was heated electrically by a bus bar arrangement fitting into the mold. A 3/8-inch pipe was supported by the casting line for the distribution of an air-excluding atmosphere, Freon No. 114. A heat lamp was placed over the Freon regulator and flow meter to prevent freezing. The mold was externally heated with four maxim gas burners. Tempilstiks were used to determine mold temperatures.
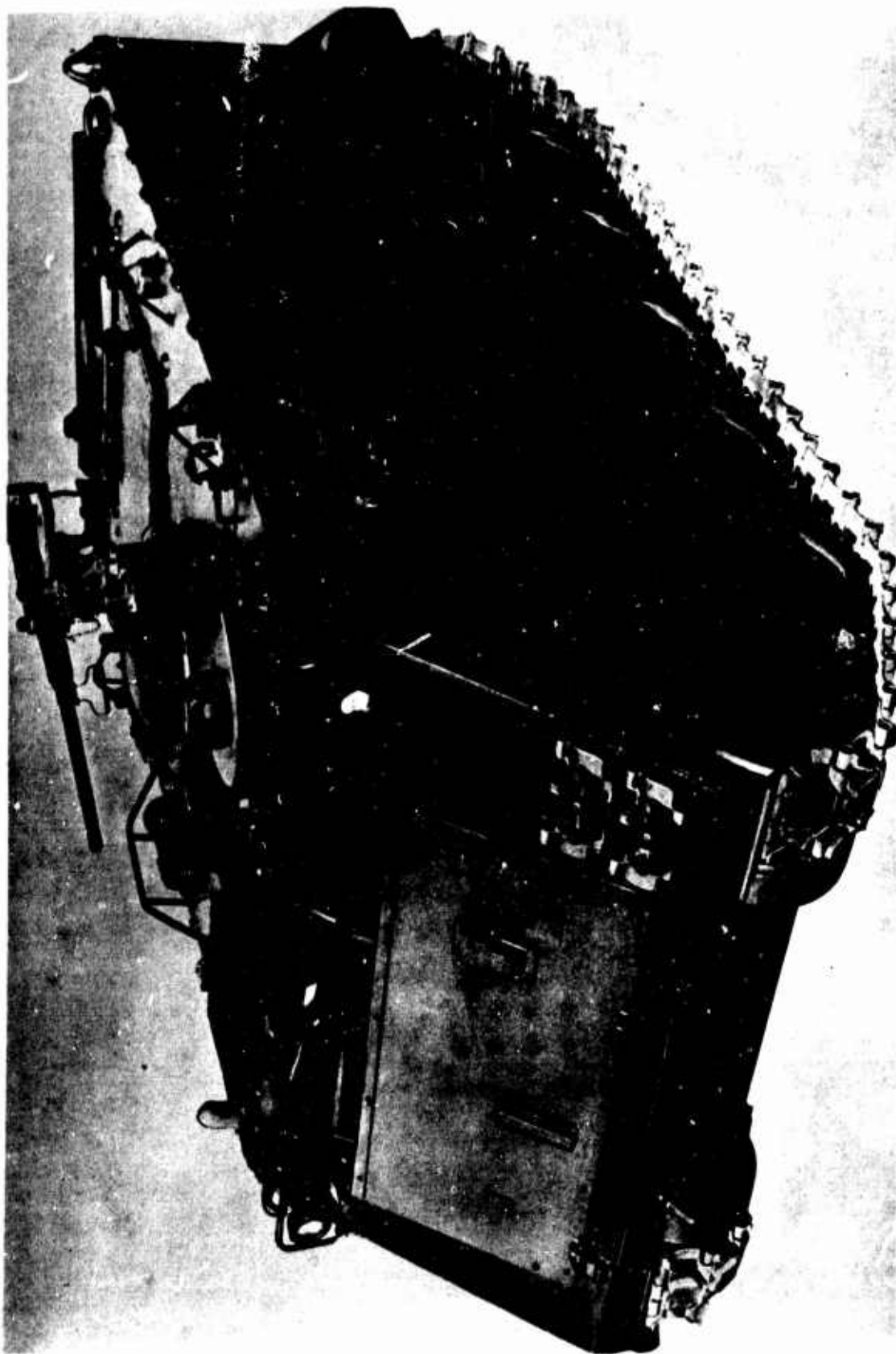
87

33325

FIGURE 36. M113 ARMORED PERSONNEL CARRIER

FIGURE 37. AS-CAST 11 x 27 x 86-INCH MAGNESIUM-
LITHIUM INGOT

Courtesy Dow Chemical Company.

Prior to casting, the mold and casting line were heated. Freon No. 114 was fed into the mold and the casting pump was started. The metal level in the mold was controlled by pump speed and rate of mold descent. While Freon No. 114 controlled burning in the mold, it did not prevent oxidation. As a result, heavy oxide skins were formed.

Dow encountered a number of difficulties in producing large magnesium-lithium alloy ingots. All the difficulties were associated with lack of homogeneity.

In an attempt to improve homogeneity, a slower casting rate was used and an air blast was directed on the mold. Both modifications were designed to produce faster solidification. Both modifications were unsuccessful. The slower casting rate resulted in lapping of heavy oxide skins. Air-blast cooling of the mold did not improve solidification rate.

As a result of the difficulties encountered in producing large magnesium-lithium alloy ingots, Dow recommended additional Government-sponsored work on the alloys. Specifically recommended was development of a process for casting high-quality large rolling slabs.

Dow's flux method incorporates some advantages over the Battelle method. Most important. Dow was able to circumvent most of the melting problems inherent in the flux process by using pumps to transfer the molten metal to the ingot mold. Casting problems, however, were not solved. Evidently, casting of magnesium-lithium alloys in air leads to formation of gross defects on the surface of ingots. These defects subsequently are associated with cracking and laminations in rolled products.

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Brooks and Perkins Method

Brooks and Perkins, Inc., Detroit, is currently the only producer of wrought magnesium-lithium alloys in the United States. This company primarily melts and casts conventional magnesium alloys. Magnesium-lithium alloys constitute less than 10 percent of sales.

The company has developed an inert-gas technique for melting and casting magnesium-lithium alloys. The crucible, 1050-pound capacity, is equipped with a closed lid and pouring spout. Prior to melt down, the crucible is pumped down to a partial vacuum and then purged with argon.

A 2-day melting cycle is used. On the first day, magnesium is melted down and allowed to solidify. On the second day, aluminum and lithium are added. This melting procedure is required because the volume of the total charge exceeds the volume of the closed crucible. The crucible is heated in a gas-fired pit furnace.

Two sizes of slab molds are available. The largest mold holds a 600-pound ingot measuring about 8 by 38 by 33 inches. The smaller mold holds a 400-pound ingot measuring about 6 by 32-1/2 by 30 inches. Both molds are made of 1-inch-thick steel plate.

Prior to pouring, the mold is heated to 400 F and purged with welding-grade argon. The melt is poured at 1200 F.

For pouring, a flexible hose is fastened to the spout of the crucible and the melt delivered to the bottom of the mold. The hose is continuously raised as the mold fills. Essentially, this process is a form of bottom pouring. Currently B&P is experimenting with the Durville pouring process.

After pouring, burning is controlled by sprinkling a fluoride-potassium-fluoroborate mixture over the exposed area.

B&P has experimented with pouring through steel wool, perforated plate, glass wool, and Fiberglas. None of these screening materials has yielded beneficial results. At the present time, none is used.

Although the B&P argon method eliminates the need for a flux, all the problems associated with melting and casting magnesium-lithium alloys in air remain. Currently, the aerospace plants using the alloys are experiencing heavy rejection rates in critical components due to dross, porosity, lamination, and other defects. In addition, difficulties in forming the alloys have been encountered. It is reasonable to assume that melting and casting the alloys under only a partial protective atmosphere is primarily the cause of current problems.

Frankford Argon Method

The Pitman-Dunn Laboratory at Frankford Arsenal has maintained an active program on magnesium-lithium alloys since the early 1950's. Early work at Frankford was connected with magnesium-lithium alloys for armor. More recently the Pitman-Dunn Laboratory has been conducting research for NASA to develop methods for producing magnesium-lithium alloy castings.

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Basically, the Frankford melting technique[13] is a closed-system argon melting and casting operation. The melting equipment, Figure 38, consists of a 75-pound-capacity gas-fired furnace. The crucible is a mild-steel pot of the type commonly used for melting magnesium. The top portion of the crucible is fitted with a steel cover plate. Figure 39 is a schematic illustration of the melting equipment.

For melting, the required weight of magnesium was charged to the crucible, which was then sealed and purged with argon for 5 minutes. A small positive argon pressure was maintained during the melting cycle. The crucible was heated to 1300 F and held at that temperature while alloying additions were made. The lithium and other alloying additions were fastened to a steel rod and pulled up into the charging tower prior to sealing the furnace. After the magnesium was molten, it was possible to plunge the lithium into the melt without breaking the argon seal.

If additional alloying were necessary, the charging-port cover was closed and the rod was plunged into the melt. Following charging, the melt was stirred vigorously and allowed to settle for 5 minutes. The metal then was reheated to the pouring temperature (approximately 1325 F) and poured into the mold which previously had been purged with argon. An argon cover was maintained during the pouring and until the metal was frozen.

Frankford has used the low-pressure gas method for transferring the molten alloy from crucible to mold. Essentially, the technique consists of pumping by applying argon pressure in the crucible. This method gives good results by minimizing turbulence during the pour and, hence, contributing to clean metal ingots and castings.

A number of mold materials[14, 15] have been used. Test bars have been cast in a split permanent mold machined from steel. Shapes have been cast in machined and in rammed graphite molds. Recently[16], the Arsenal has reported on investment casting of magnesium-lithium alloys.

The Frankford argon method incorporates several advantages over other methods used for melting and casting magnesium-lithium alloys. Argon melting eliminates flux entrapment. The argon pumping technique used to transfer the molten metal from crucible to mold minimizes turbulence and prevents direct exposure of the metal to the atmosphere. Thus, the method seems particularly suitable for production of small castings. However, it would be difficult to scale-up the process for production of commercial-sized ingots.

American Machine and Foundry Method

In 1965, the American Machine and Foundry Company (AMF) developed an inert-gas technique for melting and casting magnesium-lithium alloys. The work[17], still in progress, is being performed for the George C. Marshall Space Flight Center of the National Aeronautics and Space Administration. AMF techniques incorporate some features not previously used with these alloys.

For melting, AMF used a heavy-duty electric furnace mounted on frames for easy tilting and direct pouring. A photograph of the assembly is shown in Figure 40. This equipment was used for preparing 10-pound heats.
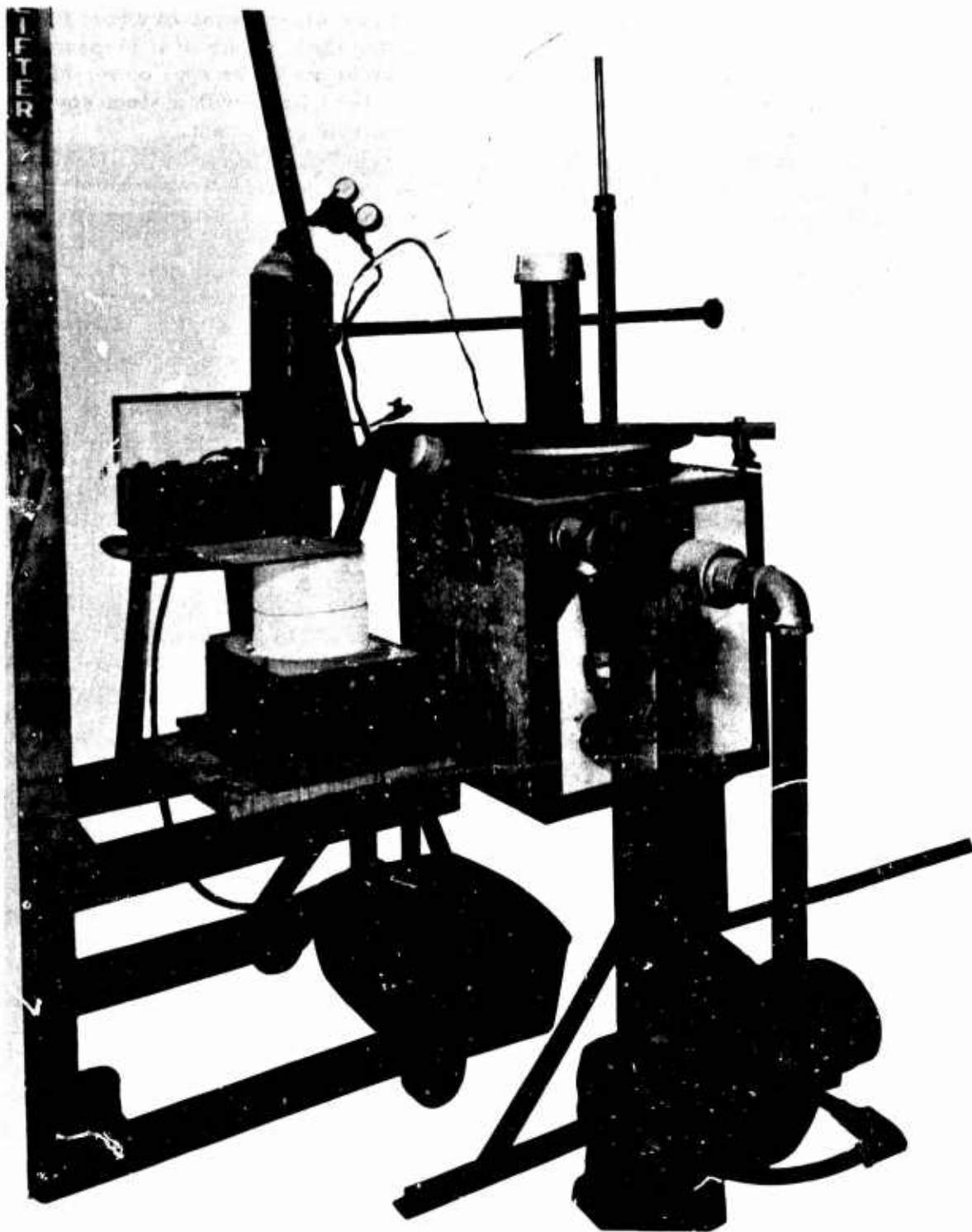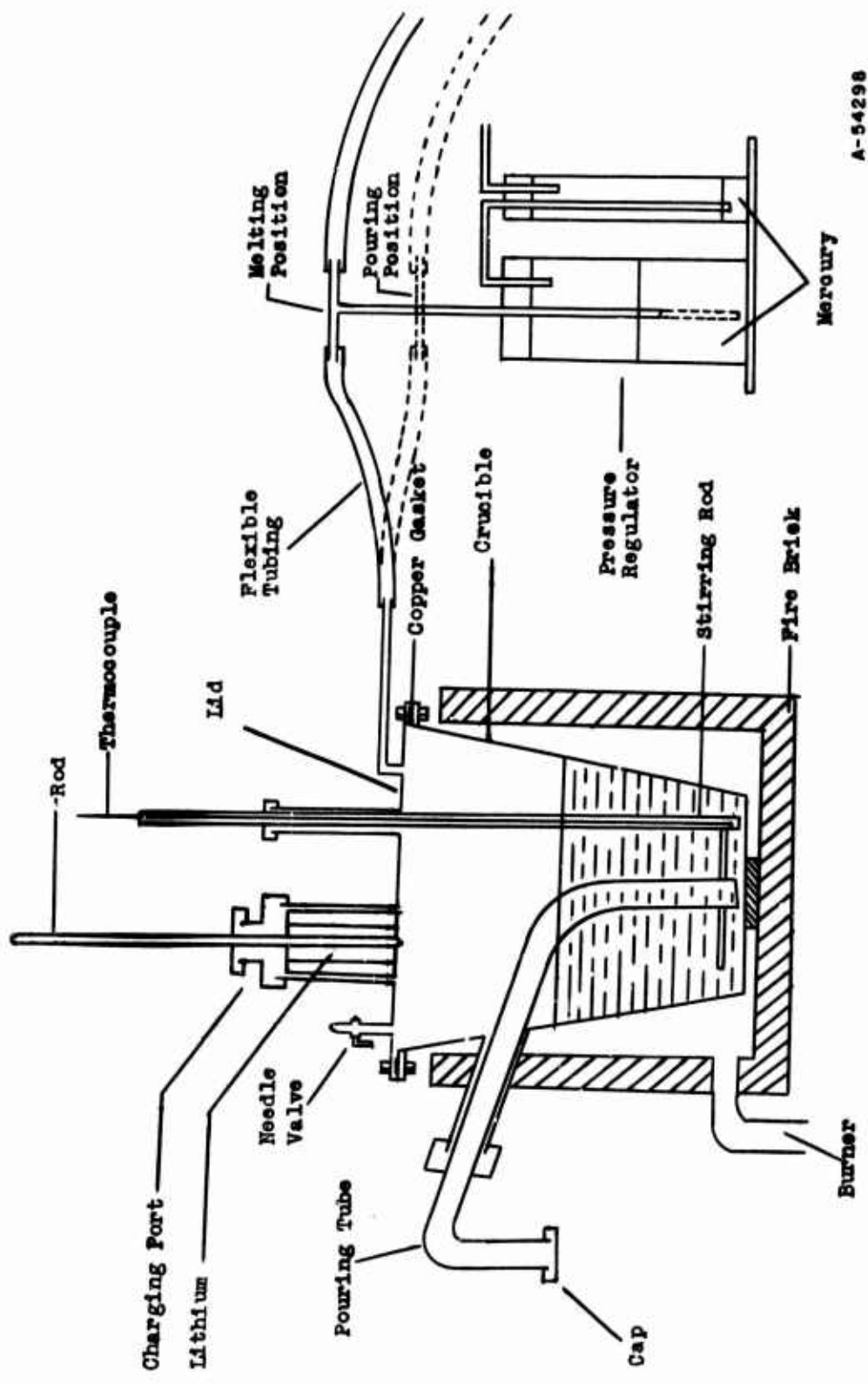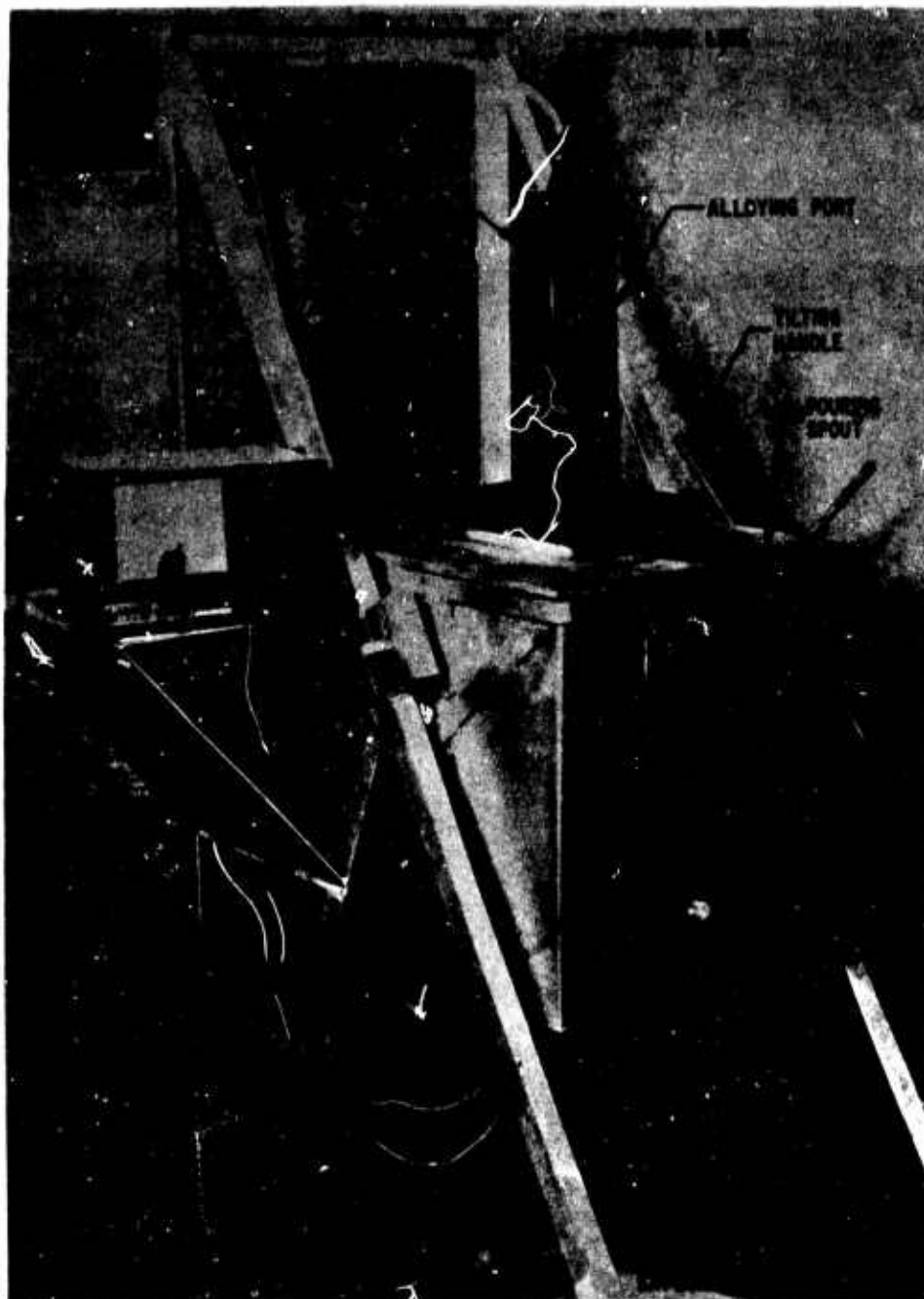
FIGURE 38.   MAGNESIUM-LITHIUM FURNACE SHOWING MOLD IN POSITION
PREPARATORY TO POURING

Courtesy Frankford Arsenal

FIGURE 39. SCHEMATIC OF MAGNESIUM-LITHIUM MELTING FURNACE

Courtesy Frankford Arsenal.

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ALLOYING PORT

TILTING HANDLE

POURING SPOUT

33328

FIGURE 40. AMF FURNACE USED TO MELT MAGNESIUM-LITHIUM ALLOYS

The crucible is equipped with a thermocouple well to which a stirring paddle is attached. Another port is used to admit argon. During the pouring cycle, the crucible was continuously flushed with purified argon and additions made to the melt through a large port located on the top of the crucible.

Prior to charging, the crucible was heated to 1350-1400 F. After reaching equilibrium, 80 percent of the magnesium was added to the crucible and the top tightly secured. After the initial charge melted, the remaining magnesium was added to the melt by lowering it through the alloying port. Additional elements were added in sequence through the same port with the grain refiner and lithium. Following zirconium additions, the melt was slowly "puddled". After the final lithium addition, the melt was thoroughly stirred. Lithium was added as a Mg + 47 percent Li master alloy, developed by the Foote Mineral Company at the request of AMF project personnel. The use of a master alloy insured thorough mixing and complete lithium recovery. This use of a Mg-Li master alloy is the first reported in the literature.

After the melt was thoroughly stirred, the pouring spout was opened and the crucible removed from the furnace so that a portion of the metal could be poured into a fracture mold. The crucible was then returned to the furnace and the pouring spout closed while the fracture casting was examined for grain refining. If the proper texture was obtained, the pouring spout was again opened and the crucible was removed from the furnace and the alloy quietly poured. The melt was either poured directly into the mold or into a hot ladle for subsequent teeming to the mold.

All the alloys were cast into a split cast-iron permanent mold designed to facilitate production of oxide-free cast rolling stock.

Five insulating and/or fluidity-promoting mold coatings were evaluated by AMF. The coatings were

(1) Acetylene or lamp black
(2) Dycoat 140
(3) Dycoat 140 + Dycoat 11
(4) Dycoat 140 + Ep. 2210
(5) Dycoat 140 + acetylene soot.

The Dycoat 140 + Dycoat 11 combination gave excellent insulating and lubricating properties, requiring little additional attention when properly applied. Coatings were applied to the mold heated to 500 F. Temperature was measured using a surface pyrometer.

Extensive care was taken to eliminate turbulence and thus minimize the formation of oxide inclusions. Turbulence was partly eliminated by choking the metal in the sprue. Any dross that did form during pouring was trapped in the sump by a tin-plated perforated metal cylinder. Fluffed steel wool was also placed inside the cylinders to cushion the liquid metal and to eliminate turbulence. For alloys containing 7 to 12 percent lithium, double screening was necessary to keep the metal clean. For these alloys, concentric perforated cylinders with fluffed steel wool inserted between the cylinders were used for additional screening.

An as-cast ingot is shown in Figure 41. This ingot was typical of small castings produced using a gating-runner-sump system.

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FIGURE 41. AMF INGOT SHOWING RUNNER AND SUMP

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More recently, AMF has scaled-up to 50-pound pilot melts. The melting facility is illustrated in Figure 42. Essentially, the crucible is similar in design to the smaller one used for preparing 10-pound heats. However, the large crucible is lifted out of the furnace with a movable overhead crane and the heat poured directly to the mold. The mold is also similar to the smaller mold used for the 10-pound heats. Currently, AMF is evaluating the quality and properties of large ingots.

In general, the AMF argon method of melting is very similar to that used at B&P's Livonia plant for producing commercial heats of LA141A. The AMF casting process, however, is different from other ingot processes used for magnesium-lithium alloys. Bottom pouring of ingots using a sprue-runner-ingate system may have advantages for eliminating surface defects and minimizing entrapment of dross.

Battelle Vacuum-Argon Method

In 1959 Battelle developed[11] a vacuum-inert gas technique for melting and casting magnesium-lithium alloys. This work was performed for the Army Ballistic Missile Agency Redstone Arsenal, Alabama (now George C. Marshall Space Flight Center). The final report on this project, published in 1960, was instrumental in developing the current interest in applications for magnesium-lithium alloys in missiles and space vehicles.

Figure 43 shows a cutaway view of the vacuum-induction unit and the steel mold and crucible arrangement used in the Battelle pilot-production of 100-pound heats This modification of the Durville casting process allowed the molten metal to run down the mold wall and greatly minimized turbulence during pouring.

In the design of the mold, provision was made for water cooling the base. Portable heaters were provided for the mold walls to allow preheating prior to casting. A resistance-heated "hot top" was also constructed that could be lowered onto the mold after pouring to reduce solidification pipe.

The melting and casting cycle employed in the vacuum-induction technique was as follows:

(1) Charge clean, dry metal to the steel crucible mounted and braced in the induction furnace. The remainder of the solid charge was placed on a Syntron vibratory feeder for later addition to the crucible.

(2) Evacuate chamber to below 10 microns of Hg pressure, and continue pump-down until the leak rate was maintained below 10 microns per minute. Back fill the chamber with argon to a total pressure of 50 or 100 mm of Hg.

(3) Heat the steel crucible to about 1200 F. Intermittent heating was necessary to avoid reaching excessively high temperatures with the existing power source. Melt-down time, including alloying from the Syntron feeder, was about 1/2 hour.

(4) Stir melt vigorously for 2 minutes, allow a 5-minute settling time, reheat to the pouring temperature, and pour into a steel mold.

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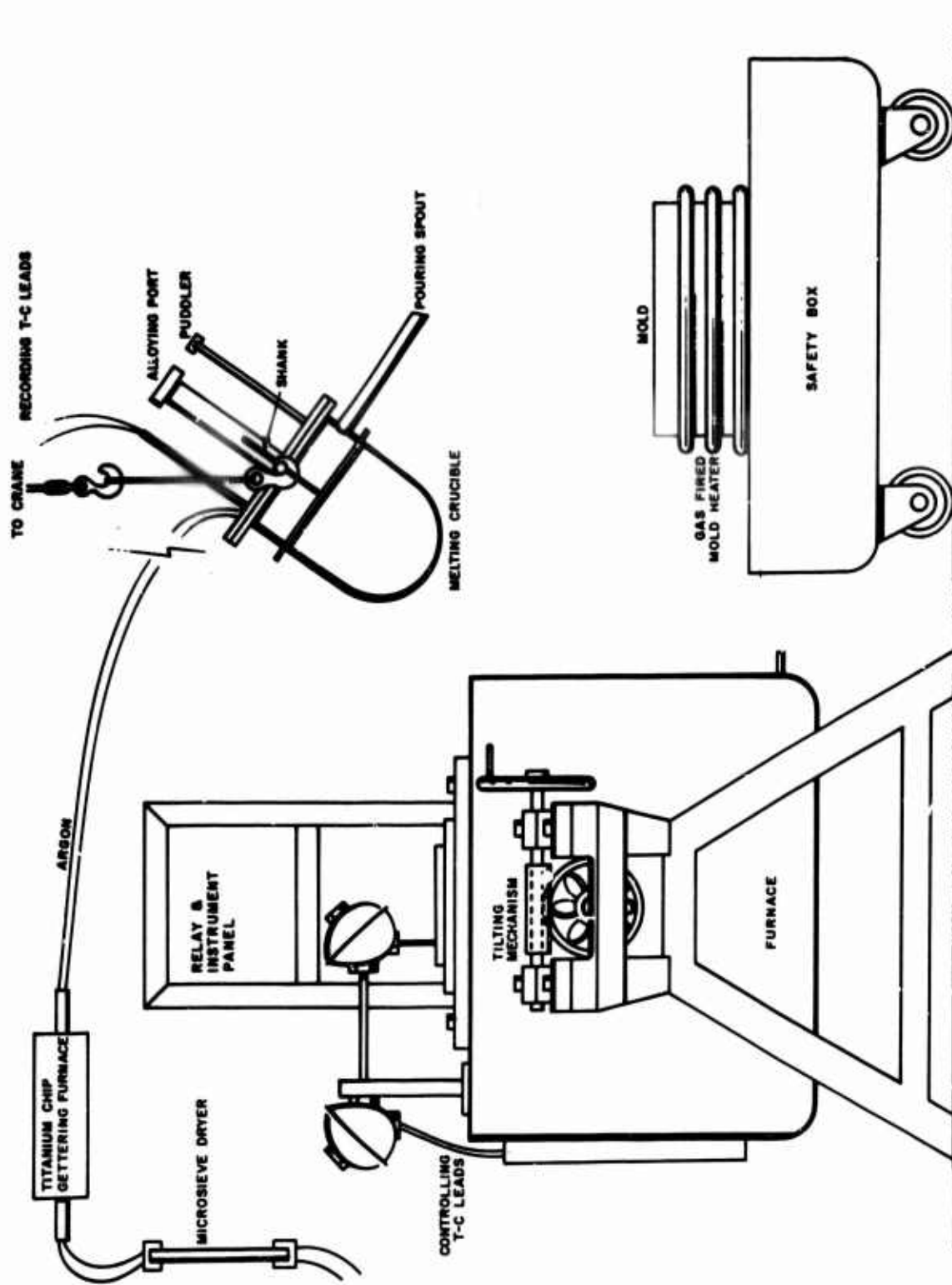
FIGURE 42. AMF MELTING AND CASTING FACILITY FOR PRODUCING 50-POUND HEATS
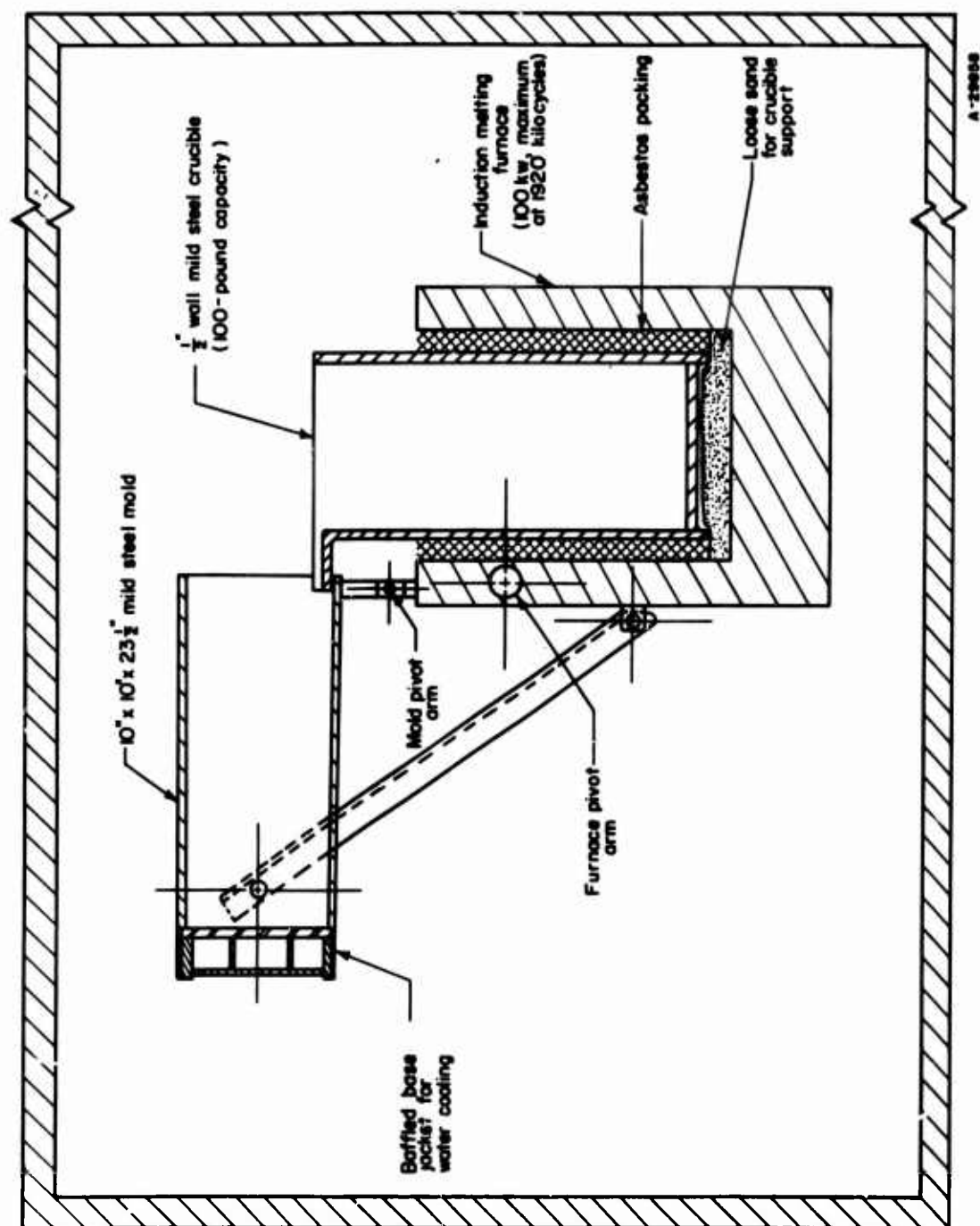
A-54299

FIGURE 43. CUTAWAY VIEW OF COMMERCIAL VACUUM-INDUCTION MELTING UNIT SHOWING MOLD AND CRUCIBLE ARRANGEMENT FOR CASTING PILOT-PRODUCTION HEATS

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Magnesium-lithium ingots cast by the vacuum-inert gas method showed very little shrinkage pipe and excellent surface quality.

This vacuum-argon method for melting magnesium-lithium alloys is the most satisfactory technique developed to date. Flux entrapment and dross formation were nearly eliminated. The process is safe and produces clean metal. However, the process as developed at that time was cumbersome. The present program for the Air Force is designed to improve the crucible and mold arrangements in order to facilitate the operation and to incorporate advantages of other melting systems if possible.

FABRICATION

Magnesium-lithium alloys, especially the commercial LA141A type alloys, are readily fabricated by almost all primary forms of metal working. These methods include rolling, extrusion, forging, and wire drawing. Currently, however, rolling is the principal method used to fabricate these alloys into sheet and plate for subsequent application. The basic fabricating procedures for the alloys were developed in the Battelle laboratories in the mid-1940's.

Dow Chemical Company reported little difficulty in rolling 1300-pound magnesium-lithium alloy ingots at their Madison, Illinois Plant. Production equipment used included an 84-inch reversing 4-high breakdown mill. Gas-fired ovens were available for heating the rolling slabs and for anneal-flattening rolled plate.

In preparation for rolling, the 9 by 25 by 66-inch slabs were heated to 450 F. These were rolled on unheated rolls to various thicknesses ranging from 4 to 1.25 inches. After rolling, the plates were anneal-flattened in an oven for 6 hours at 350 F.

Defects encountered during rolling were routed out. The routed-out areas were then filled with AZ92A magnesium weld rod, using a TIG welding unit, and then ground flush with the surface. In general, all defects, such as cracks, were traced to oxide skins and flux entrapped in the as-cast slabs.

Currently, Brooks & Perkins at their Livonia, Michigan plant are the only commercial producers of magnesium-lithium alloy (LA141A) sheet and plate. Prior to rolling, as-cast ingots are scalped to remove about 1/4 inch on each side. After homogenizing for 16 hours at 500-600 F, the slabs are rolled to the following schedule:

(1) Breakdown on heated rolls (200-300 F) using 10 to 20 percent reductions per pass

(2) Anneal 2 hours at 500 F

(3) Pickle

(4) Warm roll at 350 F to within 10 percent of finished gage

(5) Anneal and pickle

(6) Cold roll to finish gage plus 0.002-0.003 for finish pickle

(7) Stabilize 3-6 hours at 350 F under weights

(8) Cool to "hand heat" before removing weights.

Brooks and Perkins is equipped with 48-inch rolling mills. Current size capabilities are 48-inch-wide sheet at 0.050-inch thickness, 36-inch-wide sheet at 0.040-inch thickness, and 24-inch-wide sheet at 0.020-inch thickness.

The results of this survey combined with past Battelle experience indicate that heat treatment and metal working processes now available for magnesium-lithium alloys should be suitable for production of wrought products providing that quality ingots can be produced. In general, most defects encountered in fabricating magnesium-lithium alloys can be traced back to defects present in the as-cast ingot.

APPENDIX II

Mg-Li CONFERENCE

On November 9, 1966, a 1-day conference was held at Battelle with Battelle project personnel, Lt. W. T. O'Hara of the Air Force Materials Laboratory (the project monitor), and representatives of the following companies:

(1) Brooks and Perkins, Inc.

(2) Dow Chemical Company, Metal Products Department

(3) Foote Mineral Company

(4) IBM, Federal Systems Division

(5) Lithium Corporation of America.

Discussions were held concerning the following aspects of the present research program:

(1) Problem areas in the present usage of and applications for Mg-Li alloys

(2) Results obtained as of the above date on this research program

(3) Areas where assistance by representative companies would be beneficial to accomplishing the objectives of the program.

Based on these discussions, the following areas of cooperation were agreed to between Battelle and the company representatives present:

(1) Ultrasonic inspection of plate – Brooks and Perkins and IBM Federal Systems Division

(2) Supply of scrap sheet for flux studies – Brooks and Perkins

(3) Recommendations of flux compositions to be evaluated – Dow Chemical Company and Foote Mineral Company

(4) Packaging techniques for lithium metal – Foote Mineral Company and Lithium Corporation of America

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DOCUMENT CONTROL DATA - R&D

(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)

1. ORIGINATING ACTIVITY (Corporate author)	2a. REPORT SECURITY CLASSIFICATION
Battelle Memorial Institute	Unclassified
Columbus, Ohio	2b. GROUP N/A

3. REPORT TITLE

Magnesium-Lithium Alloy Casting Development

4. DESCRIPTIVE NOTES (Type of report and inclusive dates)

Final Technical Documentary Report (June 1966 – October, 1967)

5. AUTHOR(S) (Last name, first name, initial)

Byrer, T. G.   Jackson, R. J.
Frost, F. D.

6. REPORT DATE	7a. TOTAL NO. OF PAGES	7b. NO. OF REFS
December, 1967	106	16

8a. CONTRACT OR GRANT NO. AF 33(615)-5172	9a. ORIGINATOR'S REPORT NUMBER(S)
b. PROJECT NO. 9-164	AFML-TR-67-330
c.	9b. OTHER REPORT NO(S) (Any other numbers that may be assigned this report)
d.	

10. AVAILABILITY/LIMITATION NOTICES

This document is subject to export controls and transmittal to foreign governments or foreign nationals may be made only with prior approval of the Manufacturing Technology Division of the Air Force Materials Laboratory.

11. SUPPLEMENTARY NOTES	12. SPONSORING MILITARY ACTIVITY
	Air Force Materials Laboratory (MATB)
	Wright-Patterson AFB, Ohio 45433

13. ABSTRACT

The purpose of this program was to develop processing techniques for melting and casting Mg-Li alloys which would be adaptable to ingots of commercial sizes and applicable for converting them into plate and sheet. A state-of-the art report on melting, casting, and fabrication procedures for Mg-Li alloys was prepared.

Melting and casting techniques were successfully developed for the production of 50- and 100-pound ingots of LA141 alloy using a vacuum-induction melting unit under an argon atmosphere of 1-inch pressure. Subsequent evaluation of this material indicated it to be of low impurity content and to have good mechanical properties and formability

Studies of fabrication procedures showed that with high quality material, processing conditions and fabrication temperatures could vary without deleterious effects on material properties or yield. However, the standard -T7 heat treatment now used as a stabilization treatment for commercial mill products can result in subsequent hardening due to room temperature aging. Reduction of the stabilization temperature is recommended.

Studies of flux-melting techniques for the reclamation of scrap material have involved the casting of 6- 20-, and 90-pound ingots of LA141 alloy using newly developed flux compositions.

The studies showed that good quality material could be obtained by flux melting techniques and indicates considerable promise for reclaiming scrap material and ultimately reducing the cost of Mg-Li mill products.

DD FORM 1 JAN 64 **1473**

14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Magnesium-Lithium Alloys						
Light Alloy Casting						
Light Alloy Fabrication						